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This report concerns a study of the possible utilization of the mud wastes generated in the domestic production of alumina from bauxite ores. The work was done by the Battelle-Columbus Laboratories for the United States Environmental Agency. The program comprised review of technical literature published from 1940 on subjects related to technology of processing bauxite, the dewatering and impoundment of the mud residues and their possible utilization. Mud samples were received from the domestic alumina plants for characterization experiments and dewatering studies at Battelle and at the laboratories of the Dow Chemical Company in Midland, Michigan. It was concluded from the study that there is no possibility for utilization of the muds that could significantly affect the need for impoundment within the near term. However, improved mud dewatering and methods of impoundment appear possible to develop and a program of joint industry-government demonstration and pilot projects is recommended. In addition, a basic research program is recommended to study the mineral surface chemistry controlling the mechanism of dewatering. Investigations of the possible beneficiation of the muds into a raw material supplement in iron making and the possible use of mud as an absorbent in pollution abatement processes are also recommended.

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AN ASSESSMENT OF TECHNOLOGY FOR POSSIBLE UTILIZATION OF BAYER PROCESS MUDS

by

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report reviews the technology in use in the United States for production of alumina from bauxite. The purpose of the study was to assess the possibility for utilization of the mud wastes generated by the domestic alumina industry. Included in the study is a review of the published literature (1940-1975) on bauxite processing technology, waste mud dewatering, and the utilization of Bayer process muds.

This report will be of interest to alumina producers and to those individuals involved in dewatering studies of slimes and tailings produced in the minerals processing industry.

Further information on the subject could be obtained from the Industrial Environmental Research Laboratory, Office of Research and Development, U. S. Environmental Protection Agency, Cincinnati, Ohio.

David G. Stephan
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Cincinnati

ABSTRACT

This report concerns a study of the possible utilization of the mud wastes generated in the domestic production of alumina from bauxite ores. The work was done by Battelle's Columbus Laboratories for the United States Environmental Protection Agency under EPA Research Grant No. R 803760-01.

The program comprised review of technical literature published from 1940 to the present on subjects related to technology of processing bauxite, and studies on dewatering and impoundment of the mud residues and their possible utilization. Visits were made to six alumina plants in the United States to observe operations and to discuss mud-handling methods and the possibilities for utilization. Mud samples were received from the alumina plants visited for characterization experiments and dewatering studies at Battelle and at the laboratories of the Dow Chemical Company in Midland, Michigan. The Dow study was conducted under a subcontract from Battelle. A workshop was held at Battelle on March 30-31, 1976, attended by representatives from four domestic aluminum producers. Comments offered by the workshop attendees are, in part, incorporated in this report.

It was concluded from the study that there is no possibility for utilization of the muds that could significantly affect the need for impoundment within the near term. However, it appears possible to develop improved mud dewatering and methods of impoundment, and a program of joint industry-government demonstration and pilot projects is recommended. In addition, a basic research program to study the mineral surface chemistry controlling the mechanism of dewatering is recommended. Investigations of the possible beneficiation of the muds into a raw-material supplement in iron making and the possible use of mud as an absorbent in pollution-abatement processes are also recommended.

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The technology and processing of bauxite for alumina recovery is well established through more than 75 years of commercial operation in the United States and throughout the world. Within the limitations of a review study, it is not possible to assess fully the interrelationships and constraints relating to the mud disposal problem. The opportunity to visit plant operations and to discuss the study with representatives of Kaiser Aluminum and Chemical, Alcoa, Reynolds, and Martin-Marietta is therefore gratefully acknowledged. Special thanks are extended to Dr. R. M. Hansen, Kaiser, Mr. T. Galloway, Reynolds, Messrs. R. Carwile, R. Frye, and R. Newsome of Alcoa, and Mr. J. Bou, Martin-Marietta, for time spent at the plant and for providing samples for study.

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SECTION I

INTRODUCTION

The major process waste in production of alumina is the leach residue from the caustic digestion of bauxite. This waste mud is impounded to prevent the contamination of surface waters. In the United States approximately ten-million tons of mud waste is generated annually. This mud is impounded in lakes built adjacent to the refineries; mud impoundments commits land areas in the range of 2000 to 3000 acres (810 to 1215 hectares). Mud impoundment is not an ideal solution to the disposal problem. The dikes of mud lakes must be maintained, and there is always risk of a break and spill of the mud into a nearby stream or waterway. There has been little economic opportunity to date to utilize the mud.

PURPOSE

This study was made to determine whether technology is available that could make possible the economic utilization of alumina-process red- and brown-mud wastes to an extent that would eliminate the need for impoundment.

AUTHORITY

This program was initiated on June 1, 1975, by Battelle's Columbus Laboratories under U.S. Environmental Protection Agency Grant No. R-803760-01.

SCOPE

The study comprised a review of technical literature published worldwide on subjects related to technology of processing bauxite, and studies on the dewatering and utilization of red and brown muds produced during the processing. The literature review covered the period 1940 to present. Visits were made by Battelle staff to six alumina production sites to observe operations and the methods used in current disposal of the mud wastes.

Selected experiments were done to characterize red- and brown-mud samples received by Battelle from five of the six alumina plants visited. Emphasis in this experimental work was on study of the red mud derived from Jamaican bauxite because this type of mud is more difficult to dewater and presents a greater disposal problem. Laboratory studies on the use and effect of synthetic polymer flocculants to promote the settling and dewatering of the mud were made by the Dow Chemical Company under a subcontract to Battelle.

The results of the program were reviewed by Battelle with representatives of EPA and the alumina-producing industry during a workshop conference sponsored by the EPA and held at Battelle on March 30-31, 1976.

This report discusses the general background of the red-mud problem and reviews the reported research on the subject. Experimental results obtained in this program are included with Battelle's conclusions and recommendations for research to alleviate and ultimately to possibly eliminate the need for mud impoundment.

SECTION II

CONCLUSIONS

On the basis of this review, Battelle has reached the following general conclusions:

(1) Despite extensive studies made worldwide to develop means to utilize Bayer Process muds, no technology has been developed that would enable economic processing of the muds into products having sufficient existing markets to reduce the present need for impoundment of the muds generated in current domestic alumina-production operations.

(2) Virtually all potential commercial uses of the muds require that the muds be dewatered to the extent that the solids can be transported and/or stored in consolidated dry form without tendency to leach or repulp. Low-cost dewatering of the muds is therefore considered the key to their possible future utilization.

The present cost of mud impoundment ranges from $0.50 to perhaps as high as $4.00 per ton of dry solids, depending on factors such as land cost, distance of the impoundment area from plant, availability of sand and other aggregate for dike construction, rainfall, evaporation conditions, and the characteristics of the mud. Costs for future impoundment by present methods are projected to increase. It was therefore concluded that methods enabling muds to be dewatered to the conditions cited at a cost less than about $5.00 per ton of dried solids would be of interest to the industry. However, no process technology is reported to be available to do this.

(3) The chemical and physical characteristics of the Bayer Process muds, i.e., composition, particle size, and mineralogy, differ with the origin of the bauxite ore. Variations in settling and dewatering characteristics of red muds relate to these differences in the bauxite ores. Chemical factors such as pH and presence of metal ions appear to have little effect on the settling rate and ultimate settled density. Use

of flocculants can increase settling rate but offer little promise to increase ultimate settled density beyond that now achieved in mud lakes. It was therefore concluded that from the standpoint of mud disposal, little change in dewatering properties and the requirement for impoundment can be effected by changes in the conditions of operation of the Bayer Process.

(4) Chemical treatment in combination with mechanical dewatering was not found to result in a higher degree of dewatering of red mud. No improvement in ultimate solids content was achieved in tests made using magnetic flocculation, or using flocculants or coagulants in combination with centrifugation. Additional research of this type is not warranted.

(5) The physical characteristics of Bayer Process red muds may be substantially altered by energy-intensive processing such as kiln treatment, as in the case of the Combination Process used to process Arkansas bauxites. So called "brown muds" generated by the Combination Process are sintered materials of coarser particle size which settle and dewater more readily than the red muds of the more conventional Bayer Process.

(6) The addition of energy-intensive process steps to the present Bayer Process, for example, an initial reductive roasting of the bauxite, may alter its physical character and that of the mud residues in a manner analogous to the changes brought about by the Combination Process, and possibly lead to improved dewatering and a possible basis for increased recovery of metal values. However, in-plant modification of this type would probably require very substantial addition of new process equipment and major changes in present Bayer Process operations. It was concluded that relative to red-mud disposal, process-development effort that would entail substantial change in the Bayer Process flowsheet is not warranted.

(7) Installation of bottom drainage in newly constructed mud lakes appears to offer improved rates of dewatering and the opportunity to achieve high-density, stable mud solids, particularly in geographic regions with a net accumulation of rainfall. Mud lakes constructed with a bottom drainage system may develop greater permeability and a higher degree of leaching of solubles from inactive or dried mud lakes. The incorporation of bottom drainage may therefore improve the ability to grow vegetative cover on old mud-impoundment areas. Bottom drainage may also lead to improved ability to harvest dry mud which could be more economically utilized. Additional study of bottom drainage

systems and their construction, drainage and effluents, cost factors, and maintenance characteristics is warranted.

Conclusions reached by Battelle related to specific areas for possible utilization of the muds are these:

(8) Metallurgical Uses - Past interest in red mud for metallurgical purposes has been mainly as a possible source of iron for iron and steel manufacture. The production of iron from muds represents one possible use that could consume the total tonnage of mud generated by the domestic alumina industry. The muds as generated represent unacceptable material for ironmaking by conventional blast furnace or direct reduction methods. To be useful as a source of iron, the muds require processing to concentrate the iron to 55-60 percent and to substantially eliminate alumina, titania, phosphorus, and vanadium components which are considered detrimental in iron and steel making. Technology to effectively and economically do this is not presently available. Additional study to develop a nonsmelting method for concentration of iron and elimination of undesired elements for iron and steel making is considered to be warranted. Emphasis should be on production of an iron-rich material suitable for blending with other sources of iron units in iron- and steel-making operations.

To be economically viable, the cost of drying, beneficiation, and pelletizing to meet iron-making specifications, including transportation to the steel mill, must be competitive with cost of iron units as ore pellets. At present, this cost would be of the order of $50 per ton of contained iron, or equivalent to about $30 per ton of dried mud solids.

Metals other than iron and aluminum can be recovered from the muds. For example, in some cases titanium can be recovered as ilmenite or rutile concentrates by conventional gravity concentration. Removal of titanium minerals may be economical, depending on the amount and mineralogical form, but such processing has little influence on the need for impoundment of the bulk of the mud. Work on titanium-mineral separation technology is therefore not warranted. Vanadium, gallium, and scandium have also been recovered from muds in laboratory studies. Because these elements are present in very low concentration and have very limited markets, there is little merit to further development of extraction methods for these metals from the standpoint of any impact such work would have on the impoundment of alumina-process muds.

(9) Ceramic Uses - The possible use of Bayer Process muds in ceramic applications has been very extensively studied and reported. Although muds may have been tested as an additive agent in high-tonnage products such as cements, construction block mix, and lightweight aggregate, the muds have not been proven to offer any unique and beneficial properties as a raw material for these applications. It was therefore concluded that no additional general studies of use of muds in ceramic mixes is warranted.

(10) Use as a Waste Treatment Agent - Bayer Process muds have been tested and found effective as agents in treatment of both gaseous and liquid effluents. The potential merit of the use of mud for waste-treatment processing depends on location, the need to predry the mud, and the subsequent disposal procedures that will be needed. Additional study of this possible use is warranted.

SECTION III

RECOMMENDATIONS

In view of the foregoing conclusions, the following recommendations are offered.

Industry - Government Demonstration and Pilot Projects

Emphasis in the near term should be directed toward study of methods that not only will assure environmentally acceptable containment, but also offer potential for improving reclamation of impoundment areas and the possibility of harvesting the dried mud, either for commercial use or for alternative off-site disposal. Recognizing that each operating plant has different conditions and constraints in disposal related to bauxite sources, geography and hydrology of the site, land availability, and other factors, it is recommended that a series of demonstration or pilot projects be initiated in collaboration with EPA and interested individual operating companies on the site of the company. Such demonstration projects could be of sufficient general interest to the industry and to the public benefit to warrant EPA program funding on a cost-sharing basis. Specific topics considered pertinent by Battelle as subjects for such EPA-industry cooperative demonstration projects at specific operating sites are:

(1) Impoundment area drainage systems, construction, and cost factors

(2) Development of methods to achieve vegetative cover over inactive disposal areas to include study of flora that could tolerate high levels of sodium ion

(3) Study of methods to prevent airborne dust from dried mud areas

(4) Factors in the removal of dried mud solids for alternative storage or use

(5) Ability to reclaim mud lake areas for alternative uses.

Such demonstration projects should be conducted by the operating company and the activity reported to and monitored by the EPA. Award of funding support for such programs should be based on proposals submitted by the operating company to the EPA. An estimated budget believed to be warranted for demonstration projects is of the order of $500,000 during a 3-year period with cost sharing at a 50 percent Government-industry basis.

Research and development efforts considered by Battelle to have merit for possible longer range improvement and which warrant Government support are:

Basic Research on Physical-Chemical Mechanisms of Red Mud Dewatering

The inherent difficulty in developing chemical agents or apparatus to dewater the muds is related to the extreme hydrophilic tendency of the clay-like minerals comprising the structure of many of the bauxites and muds. Despite extensive research and development by the alumina industry worldwide on various chemical and mechanical methods of dewatering muds, a relatively simple or rapid and economic means to achieve a relatively high degree of dewatering of Bayer Process muds has not been found. Because of the importance of improving technology to possibly eliminate the need for impoundment, it is recommended that a research effort be established to study the mineral surface chemistry of various red muds and to better understand the mechanisms which limit and/or control the removal of interstitial and bound water by physical-chemical methods. This research should include detailed study of the mineralogic structure of various muds, using a combination of optical and physical-chemical analyses to examine the microstructure and nature of the surfaces and their affinity for water. The physical chemistry of the response of mud surfaces to various flocculants, floc strength, use of chelating agents, and possible chemical adsorbents for water should be part of a broad basic program on dewatering fundamentals and mechanisms.

An estimated budget of $150,000 covering a period of 2 years is recommended.

Red-Mud Utilization

Pertaining to possible future utilization of mud residues, Battelle recommends the following studies:

Metal Recovery–

Red muds contain a significant amount of iron and could be developed as a source of iron units in the production of iron and steel. Emphasis should be on technology for beneficiating the mud into a usable iron-ore supplement to blast furnace charge ore. For this purpose, the beneficiated mud would not be required to meet the rigid specifications placed on blast-furnace feed ore. A metallurgical process that could offer merit as a beneficiation procedure is the segregation roast method that has been applied commercially for extraction of copper and nickel from oxide ores. Segregation roasting employs a reductive chloride roast conducted with solid carbon present in the charge. In situ volatilization and reduction occur under proper roasting conditions to yield a roaster calcine containing coalesced iron adhering to the surface of the carbon particles in the charge. Iron can be removed relatively free of contaminants by usual magnetic-separation methods.

It is recommended that initial laboratory study be made of the technical feasibility of the application of the segregation roasting method for beneficiation of selected red muds.

This program can be completed within 12 months at an estimated cost of $50,000.

Adsorbent Material for Pollution Abatement--

Red and brown muds could be used as adsorbents in removing sulfur oxides from flue gases and for other gaseous- or liquid-effluent treatments. The ability to leach and recover other metal values from sulfated muds, and the factors involved in the disposal of the sulfated and/or leached mud adsorbent are of sufficient interest to warrant investigation.

To properly evaluate this potential use, it is recommended that system studies be made, including development of data on the structural strength of various dried muds to determine whether muds can be used as adsorbent without need for prilling or pelletizing. The extent of sulfur oxide adsorption of various muds and effect of different conditions of temperature should be evaluated.

A systematic study of the possible use of mud residues as adsorbents is estimated to require 18 months to complete at a cost of approximately $75,000.

SECTION IV

TECHNICAL BACKGROUND

GENERAL

Aluminum metal and aluminum oxide products are made almost entirely from bauxite ore. Bauxite is a naturally occurring mineral composed principally of a mixture of one or more of the hydrated aluminum oxide minerals gibbsite ($Al_2O_3 \cdot 3H_2O$), boehmite ($Al_2O_3 \cdot H_2O$), and diaspore ($Al_2O_3 \cdot H_2O$) and impurities of silica, iron oxide, titania, and other elements in trace amounts. It is generally believed that bauxite deposits result from intense chemical weathering of aluminum-bearing rocks or formations under tropical or subtropical conditions with alternating wet and dry seasons. Depending on the source location, the texture of the ore may be fairly granular or it may contain or be essentially all extremely fine-grained claylike material.

Alumina is extracted from bauxite using the Bayer Process. The initial step is to partially digest the ore in a caustic solution. The caustic reacts with the major part of the aluminum oxide content of the bauxite and causes it to dissolve. This reaction is specific to the aluminum oxide and some reactive silica; however, unreactive silica (i.e., quartz), iron, and titania minerals are not dissolved to any significant degree and remain as a solid residue. The residue is a solid waste which is washed to recover the active solution and then discarded. This mud material is generally extremely fine and difficult to filter. It has been least costly to dispose of this waste by pumping it as a relatively dilute suspension to holding ponds and lakes constructed for that purpose. Sufficient pond area is provided to allow the mud to settle and for clear water to be recycled to the process. Because of the iron oxide content of the waste, the waste has a red color and has been termed "red mud"; the impoundment areas are called "red-mud lakes".

From the standpoint of effluent control, impoundment of the red mud is a practical method which has been used for many years by the industry. With proper pond construction and adequate pond holding area, the red-mud waste can be retained and contamination of surface waters can be virtually eliminated.

Impoundment is not an ideal solution to the red-mud disposal problem, however, for many reasons. Red-mud lakes require a significant amount of land. The settling rate of solids is generally slow. Certain muds are almost thixotropic in character and even the old, apparently dried lakes cannot support heavy equipment or construction. These ponds, in fact, must be maintained even after they are no longer in use, and always there is some risk that a break or spill will occur and allow mud into a nearby stream or waterway.

The problems, of course, vary with the location. Availability of land is a most important factor. Moreover, commitment of the land for mud disposal may impact on land-use plans in the region. Rainfall and seasonal weather conditions play a significant role. Long periods with little rainfall cause the mud lakes to dry on the surface and become a source of windblown dust. Long periods of heavy rainfall increase the risk of a break in the dikes or can cause runoff from the pond into surface waters.

The question of whether technical developments can be made to provide practical improvements in or alternatives to present impoundment is not simple to answer. Alumina refineries are major complexes with many interrelated operations and, therefore, considerable constraint in operating conditions. Major changes in processing would be difficult and costly to implement. Ideally, such changes would provide economic benefit to the processor through recovery of additional values or reduced overall cost in disposal of the muds. It is, therefore, important to consider the structure of the domestic industry, the Bayer Process, present impoundment methods, and the probable future costs for impoundment.

THE DOMESTIC ALUMINA INDUSTRY

Bauxite ore is refined into alumina in nine refineries in the United States.* These process plants are owned by five primary aluminum producers. The location of the refineries, countries of origin of bauxite used, annual production, and waste (mud) produced are shown in Table 1. Most of the bauxite used in the United States is imported. Jamaica and Surinam are the principal suppliers. The only commercial bauxite deposits in the United States are located in Arkansas.

The amount of mud produced in processing differs for the types and ranges of bauxite and ranges from 0.3 ton (Surinam bauxite) to 2.0 ton (Arkansas bauxite) per ton of alumina produced. The domestic alumina plants generate about 10 million tons of mud waste annually.

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*The term domestic industry is intended to include the refinery in St. Croix, Virgin Islands.

TABLE 1. LIST OF DOMESTIC BAUXITE REFINING PLANTS

Company	Plant location	Type of bauxite used	Approximate plant capacity(a), metric tons alumina/year	Approximate waste (mud) produced(a), metric tons/year
Aluminum Company of America (Alcoa)	Mobile, Alabama Point Comfort, Texas	Surinam-African Caribbean-Surinam-African-Australian	1,500,000 1,100,000	450,000 1,100,000
Aluminum Company of America (Alcoa)	Bauxite, Arkansas	Domestic (Arkansas)	225,000	450,000
Kaiser Aluminum and Chemical Corp.	Baton Rouge, Louisiana Gramercy, Louisiana	Jamaican Jamaican	1,000,000 800,000	1,000,000 800,000
Reynolds Metals Co.	Corpus Christi, Texas Hurricane Creek, Arkansas	Jamaican-Haiti Domestic (Arkansas)	1,300,000 755,000	1,300,000 1,510,000
Ormet Corporation	Burnside, Louisiana	Surinam	560,000	168,000
Martin-Marietta Aluminum Co.	St. Croix, Virgin Islands	Weippa-Guyana-Surinam-Kassa-Boke (Guinea)	325,000	--

(a) Information provided by individual operating company.

Bauxite

Alumina is produced domestically only by extraction from a bauxite ore mineral. The term bauxite refers to an ore or mixture of minerals formed by the weathering of aluminum-bearing rocks. Bauxite deposits are found throughout the world. Bauxites are composed principally of a mixture of one or more of the hydrated aluminum oxide minerals. Gibbsite (also called hydrargillite) is a trihydrate form of alumina ($Al_2O_3 \cdot 3H_2O$). Boehmite and diaspore are two forms of alumina monohydrate ($Al_2O_3 \cdot H_2O$). The relative amount of each form present in the bauxite is very important because the trihydrate is more readily extracted and milder digestion conditions can be used.

Other aluminum oxide minerals generally contained in bauxites include the hydrated aluminum silicate minerals halloysite and kaolinite. The associated iron minerals are goethite and hematite. Titanium is often present in the minerals rutile, anatase, and ilmenite. Silica is present in quartz and clays. The form and extent of silica contained are very important in the economics of alumina extraction. Silica in the form of quartz is only moderately attacked during the extraction of bauxite; however, silica in kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) dissolves in the caustic (soda) causing a loss of soda and loss of alumina through formation of insoluble sodium aluminum silicate ($3Na_2O \cdot 3Al_2O_3 \cdot 5SiO_2 \cdot nH_2O$). Soda represents a significant investment and its recovery makes the process economical. Other elements are generally present as indicated in Table 2. It might be noted that bauxites used by the domestic industry are relatively low in the monohydrate minerals. Monohydrate ores are processed more commonly in Europe.

PROCESS TECHNOLOGY

The domestic alumina industry uses two basic processes for alumina production. Imported bauxites are treated by the Bayer process. The Arkansas bauxites are processed by a "Combination Process" which incorporates a sinter step with the Bayer method.

The Bayer Process

The Bayer Process for extracting alumina from bauxite was invented by Karl Josef Bayer in Austria in 1888. This process has been used in the U. S. since 1894.

The basic principle involved in the Bayer Process is to dissolve the aluminum component of bauxite ore in caustic soda solution. The solution is first treated to remove impurities such as silica and iron, and is then conditioned to precipitate alumina trihydrate. This material produces aluminum oxide (alumina).

TABLE 2. CHARACTERISTIC ANALYSES OF VARIOUS BAUXITES[a]

Constituents	Weight percent Jamaican	Surinam	Arkansas	Guyana
Al_2O_3, total	49.2	55.0	48.7	58.6
SiO_2	0.7	3.8	15.3	4.9
Fe_2O_3	19.3	7.0	6.5	4.1
TiO_2	2.5	2.4	2.1	2.5
F	--	--	0.2	0.02
P_2O_5	0.4	0.06	--	--
V_2O_5	0.03	0.04	--	--
H_2O, combined	26.5	31.2	25.8	29.6
Al_2O_3, trihydrate	44.4	50.0	34.1	52.7
Al_2O_3, monohydrate	2.8	0.2	14.6	5.9

(a) Analyses provided as typical by the operating company.

A generalized flowsheet of the Bayer Process is shown in Figure 1. The first step in the process is to grind or break up the nodules of bauxite. The bauxite is then slurried with a portion of the recycle caustic soda solution (spent liquor) and pumped to large pressure vessels (digestors) where it is mixed with the main stream of spent liquor and heated with live steam. The temperature and pressure required for alumina digestion depend on the relative amount of monohydrate and trihydrate in the ore. For a predominantly monohydrate bauxite, a temperature of 200 to 250 C (392 to 482 F) and a pressure of about 35 atmospheres (500 psi) would be used; for trihydrate, a temperature of 120 to 170 C (248 to 338 F) and a pressure of 3 to 5 atmospheres (50 to 70 psi) are used. The operating conditions are also adjusted depending on the ratio of monohydrate and trihydrate alumina forms. The digestion reactions form soluble aluminate by the following reactions:

$$\text{(Monohydrate)}\ Al_2O_3 \cdot H_2O + 2NaOH \rightarrow 2NaAlO_2 + 2H_2O$$

$$\text{(Trihydrate)}\ Al_2O_3 \cdot 3H_2O + 2NaOH \rightarrow 2NaAlO_2 + 4H_2O.$$

The solution bearing the dissolved aluminum component of the bauxite (green liquor) is sent to a "flash" heat recovery operation, where both the elevated temperature and pressure are reduced to near atmospheric conditions. The heat recovered from the green liquor as flashed steam is used in heat exchangers to preheat the solution going into the digestor. From the flash tanks, the slurry is sent to thickeners where the suspended undissolved solids are flocculated (usually with a starch) and removed in the thickener underflow. The separated mud is washed by countercurrent decantation to recover dissolved alumina and caustic. The mud in the thickener underflow is pumped at 15 to 22 percent solids to the mud-lake-impoundment areas.

The separation and washing of mud from the pregnant liquor can also be done by filtration. The use of filters depends on the characteristics and the amount of mud produced.

The thickener and washer overflow (green liquor) is sent to the pressure filter (Kelly filters) for removal of fine solids. The green liquor is then supersaturated with respect to the dissolved alumina content by a further cooling operation (vacuum flash). The liquor is then sent to precipitators, where fine alumina (known as seed) is added to enhance trihydrate alumina [$Al(OH)_3$] precipitation. The settled coarse particles of alumina trihydrate are washed, filtered, and calcined in a kiln to yield the calcined alumina (Al_2O_3) product. The spent liquor from the primary classification is sent to secondary and tertiary classification to recover fine alumina that is used as the seed material in the classifiers. The spent liquor from the final classification is sent to vacuum evaporators for concentration and is then recycled to the digestion process. A certain amount of caustic is lost in processing and this is made up either through direct addition of caustic,

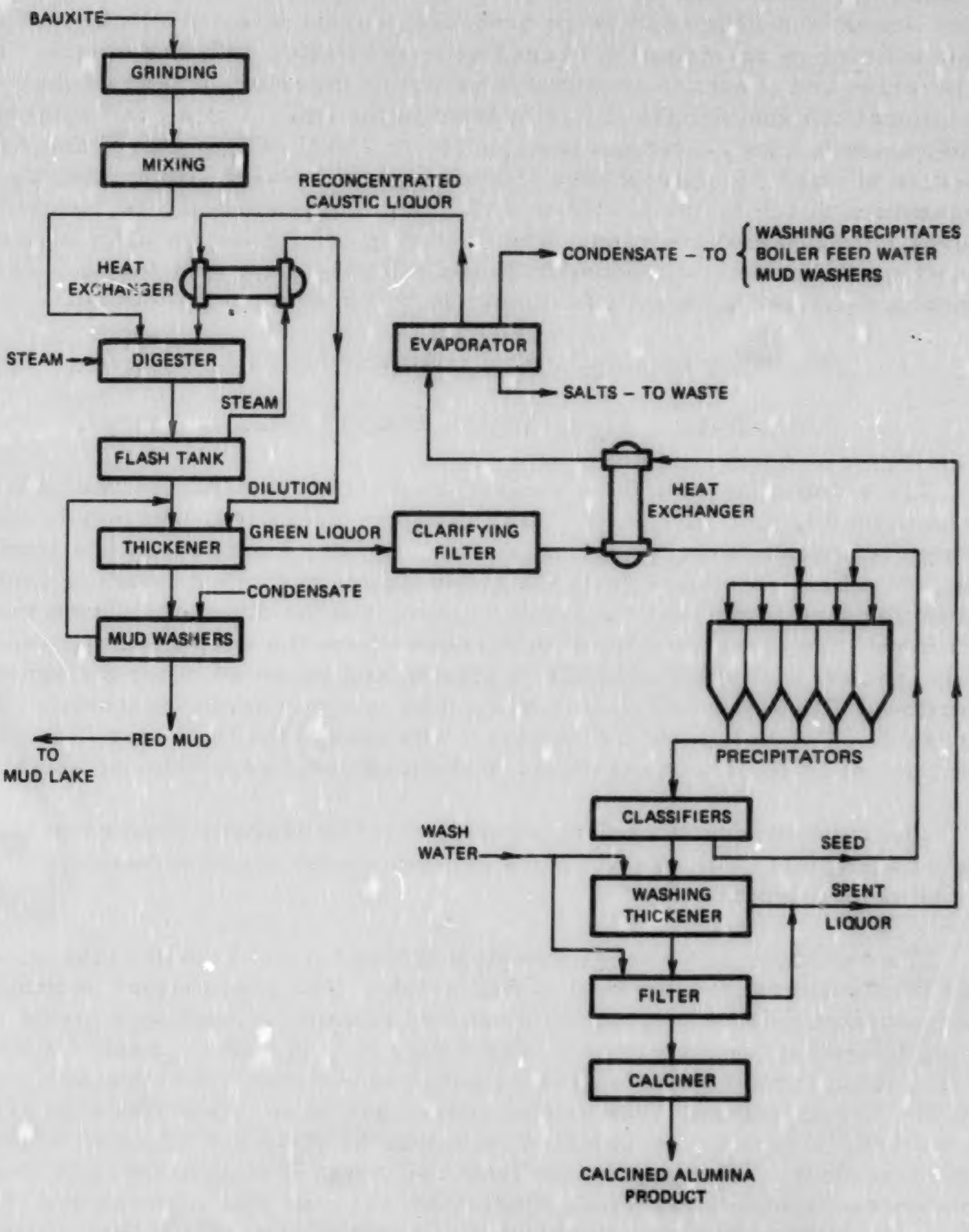

BAUXITE
GRINDING
MIXING
RECONCENTRATED CAUSTIC LIQUOR
HEAT EXCHANGER
CONDENSATE – TO
WASHING PRECIPITATES
BOILER FEED WATER
MUD WASHERS
EVAPORATOR
STEAM
DIGESTER
SALTS – TO WASTE
STEAM
FLASH TANK
DILUTION
HEAT EXCHANGER
THICKENER
GREEN LIQUOR
CLARIFYING FILTER
CONDENSATE
MUD WASHERS
PRECIPITATORS
RED MUD
TO MUD LAKE
CLASSIFIERS
WASH WATER
SEED
WASHING THICKENER
SPENT LIQUOR
FILTER
CALCINER
CALCINED ALUMINA PRODUCT

Figure 1. Generalized flow diagram of the Bayer Process.

through use of soda ash, or by use of hot caustic solution for descaling of various items of equipment such as the digestors, flash chambers, and classifiers.

The Combination Process

The Combination Process is a modification of the Bayer Process developed for bauxite containing high amounts of silica, such as those from Arkansas. The digestion of this type of bauxite by the conventional Bayer method would result in excessive loss of alumina through formation of insoluble sodium aluminum silicate. The Combination Process, also known as the sinter/leach process, recovers the alumina values that become trapped in the Bayer red mud as silicates. This additional extraction step is accomplished by mixing the red mud from the Bayer circuit with limestone and sodium carbonate and then sintering the mixture in a kiln at 1100 to 1200 C (2030 to 2192 F). The kiln product is ground and leached to produce additional sodium aluminate solution which is filtered and is either added to the main stream for precipitation or is precipitated separately. Precipitation of aluminum trihydrate is done as described previously for the Bayer Process. The washed residual solids (brown mud) consisting mainly of dicalcium silicate are pumped to a mud lake. A generalized flow diagram of the Combination Process is shown in Figure 2.

One feature of the Combination Process is that lime and soda ash substitute totally for caustic soda as the starting agent, utilizing the familiar causticizing reaction:

$$Ca(OH)_2 + Na_2CO_3 \rightarrow 2NaOH + CaCO_3.$$

During sintering of red mud with lime and soda ash, the important reaction is the conversion of silica to calcium silicate and residual alumina to sodium aluminate:

$$\underbrace{Na_2O\cdot Al_2O_3\cdot 2SiO_2}_{\text{Red Mud}} + 2CaC + 2H_2O \rightarrow 2NaAlO_2 + \underbrace{2CaO\cdot 2SiO_2\cdot 2H_2O}_{\text{Brown Mud}}$$

Mud Disposal

Solid wastes are generated in virtually all mining and processing of hard rock minerals. Mineral process wastes are generally impounded in tailings ponds. Alumina production is no exception. The mud-lake impoundment is the lowest cost method for containment of these muds from alumina production. Because of the enormous tonnage of solids generated and their near-colloidal character, these lakes present special problems in construction and maintenance. It is pertinent to consider present mud disposal and the cost

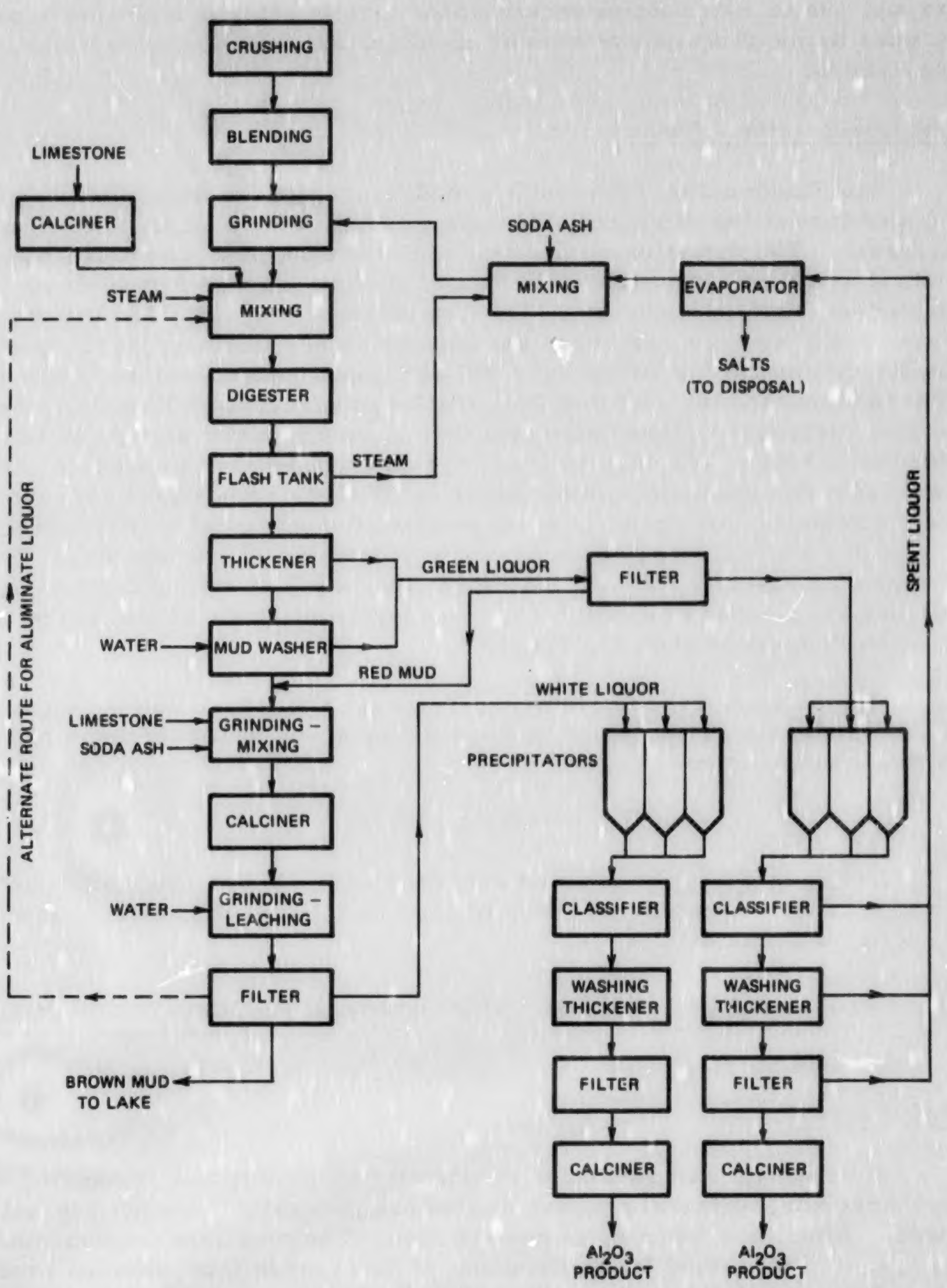

CRUSHING
BLENDING
LIMESTONE
CALCINER
GRINDING
SODA ASH
MIXING
EVAPORATOR
STEAM
MIXING
SALTS
(TO DISPOSAL)
DIGESTER
FLASH TANK
STEAM
ALTERNATE ROUTE FOR ALUMINATE LIQUOR
SPENT LIQUOR
THICKENER
GREEN LIQUOR
FILTER
WATER
MUD WASHER
RED MUD
WHITE LIQUOR
LIMESTONE
SODA ASH
GRINDING – MIXING
PRECIPITATORS
CALCINER
WATER
GRINDING – LEACHING
CLASSIFIER
CLASSIFIER
WASHING THICKENER
WASHING THICKENER
FILTER
BROWN MUD TO LAKE
FILTER
FILTER
CALCINER
CALCINER
Al_2O_3 PRODUCT
Al_2O_3 PRODUCT

Figure 2. Generalized flow diagram of the Combination Process.

factors involved in evaluating possible alternative uses for the mud and costs likely to be required to convert the mud into usable forms.

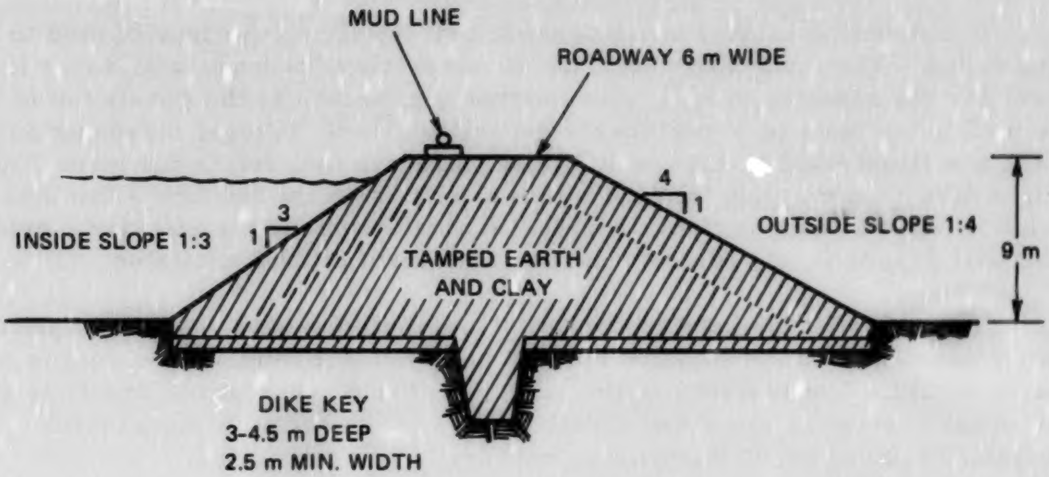
Construction of mud lakes depends principally on the type of mud to be impounded. The mud-lake dikes for the more claylike muds will differ from those for the sandlike muds. The general approaches to the construction of the mud lakes have been previously described.(1,2) Typical construction plans are illustrated in Figure 3. These differ in that one is for initial full-height dike construction, while the other calls for first building a low dike which is later raised as the lake fills. A final construction plan for a mud lake will depend on the type of mud and the extent of land available.

The cost of mud-lake construction and mud disposal can vary significantly depending on the location and the nature of the mud. Land cost is a major factor. The distance of the lake from the plant is important as is the net rainfall/evaporation rate. Available costs data were in the general range of $1.00 to $5.00 per metric ton (dry) of mud disposed.

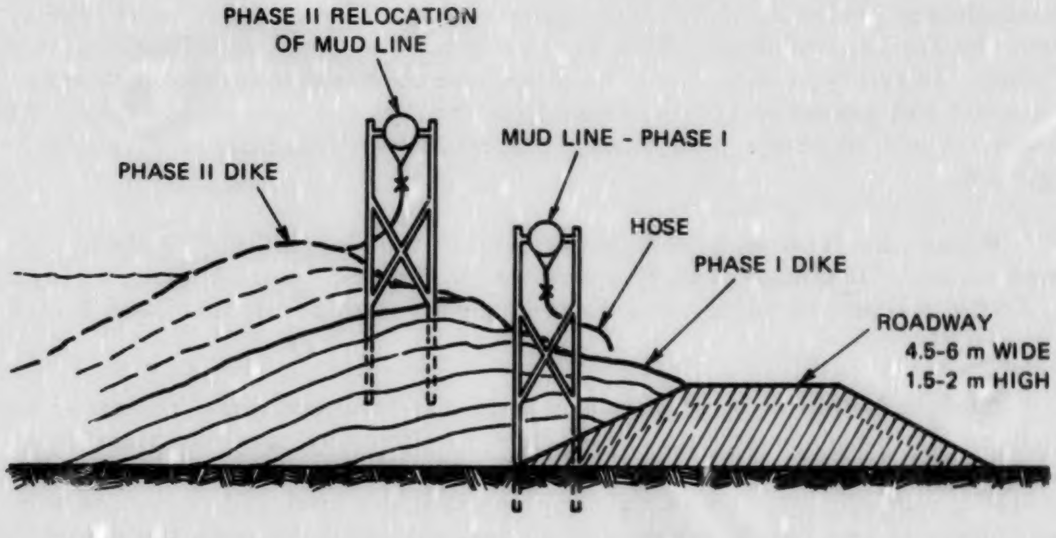
The Kaiser Aluminum and Chemical Corporation has reported studies(3,4) made to develop improved methods for impoundment of red muds generated by processing Jamaican bauxite at Kaiser's plants in Baton Rouge and Gramercy, Louisiana. On the basis of results from laboratory and pilot impoundment projects, Kaiser has constructed a 200-acre red-mud lake to serve the Gramercy plant. This lake incorporates sand-bed filtration of the "French Drain" type with drainage pipes and covering layers of different size sand and gravel to give permeability through the base of the lake. The placement of a drainage pipe during construction of the lake is shown in Figure 4.

Kaiser has termed the disposal system the Deep DREW System (Decantation, DRainage, and Evaporation of Water). A mud lake employing the DREW System is being constructed by Kaiser to serve its Baton Rouge refinery.

The mud-disposal program and pilot development work by Kaiser has provided a basis to anticipate a reduction in slurry volume contained in a lake with bottom drainage to about one-fourth the volume in the conventional "as-is" impoundment. Kaiser anticipates that the mud will consolidate in these lakes to an overall average of 50 percent solids through the combination of drainage and evaporation. It is believed, based on demonstration plots, that the lakes will dry to sufficient stability to support men and equipment. Thus, the dried lakes might be periodically surface mined to remove a substantially dry mud that would be a more economical source for iron values or for other purposes than the wet muds.

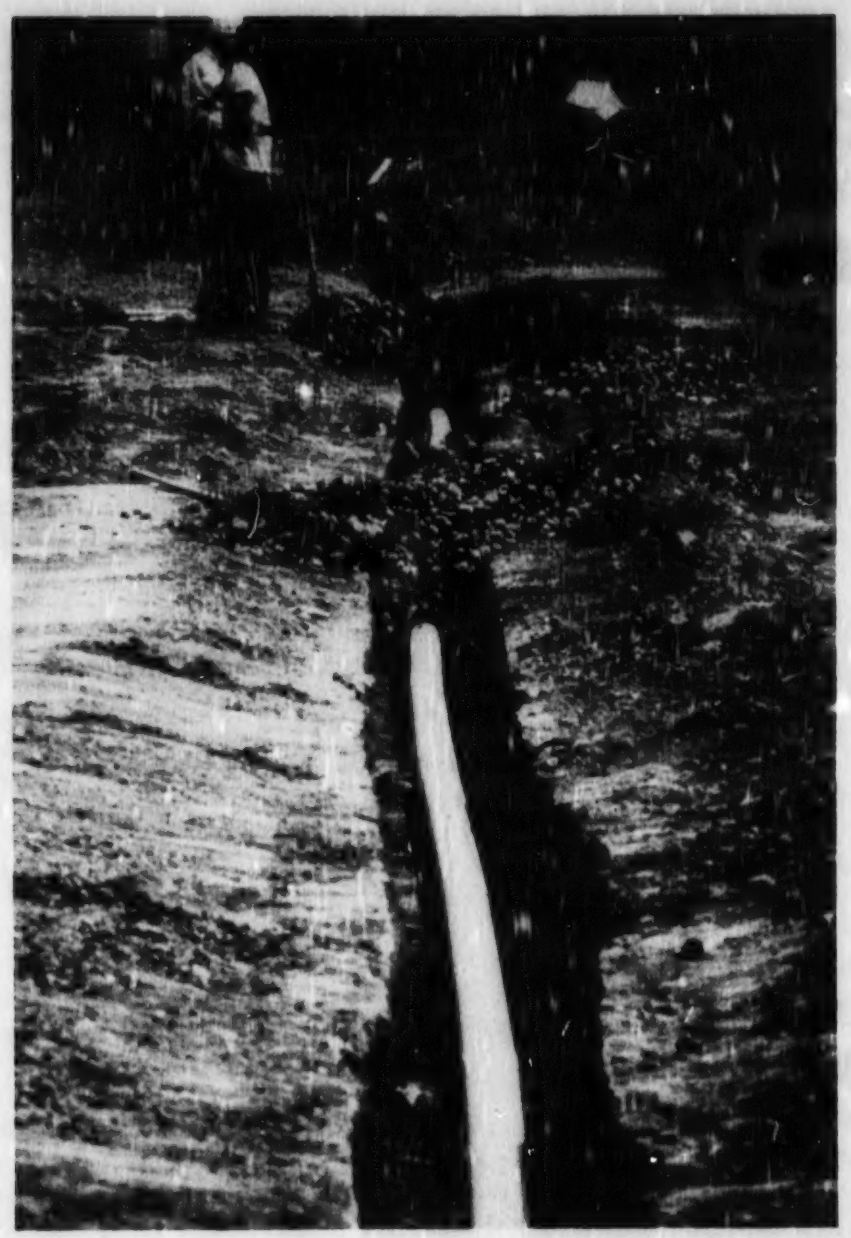

MUD LINE
ROADWAY 6 m WIDE
3
1
INSIDE SLOPE 1:3
4
1
OUTSIDE SLOPE 1:4
9 m
TAMPED EARTH AND CLAY
DIKE KEY
3-4.5 m DEEP
2.5 m MIN. WIDTH

A. INITIAL FULL DIKE CONSTRUCTION


PHASE II RELOCATION OF MUD LINE
PHASE II DIKE
MUD LINE - PHASE I
HOSE
PHASE I DIKE
ROADWAY
4.5-6 m WIDE
1.5-2 m HIGH

B. BUILDUP CONSTRUCTION OF MUD LAKE DIKE

Figure 3. Mud lake dike construction.



A system to permit use of red mud tailings as land fill instead of discharging them into the Mississippi River is under construction at a Louisiana alumina plant. Here drainage pipe is placed in a trench at bottom of storage pond; later it will be covered with coarse permeable gravel bed.

Kaiser Aluminum & Chemical Corporation photo

Figure 4. Construction of red-mud lake based on gravity drainage system. (Reprinted by special permission from Miller Freeman Publications, Inc.)

PLANT TRIPS

Visits were made by Battelle staff to review the refinery practices and methods used in mud disposal and to arrange for acquisition of mud samples for laboratory study. The plants visited by Battelle were:

(1) Alcoa - Point Comfort, Texas
(2) Alcoa - Mobile, Alabama
(3) Alcoa - Bauxite, Arkansas
(4) Reynolds Metals - Hurricane Creek, Arkansas
(5) Kaiser Aluminum - Gramercy, Louisiana
(6) Martin-Marietta - St. Croix, Virgin Islands.

These plants were selected because each used a different ore source and the mud-disposal requirements for each were different. Except for the Arkansas operations, the Bayer Process was in use. Because of the high silica content of the Arkansas bauxite, the Combination Process is used for this ore.

During the visits to the plants, discussions were held with company personnel concerning factors related to alumina production and the mud problem. The salient points were as follows.

(1) The key factor is the nature of the bauxite used. Both the quantity of mud produced and the ability to wash and dewater the mud depend almost entirely on the bauxite. The quantity of mud obtained from processing a sandy-type bauxite is generally substantially less per ton of alumina made. Sandy bauxites usually contain hematite as the predominant iron mineral and can be filtered and more easily washed.

(2) Within practical limits of variation of the process conditions, the chemistry of the process has little influence on the nature of the mud or its dewatering characteristics.

(3) Transportation of the mud from the plant to the impoundment lake is done most conveniently and economically by pumping as a slurry. The practical limit of the solids content of such a slurry is 25 to 40 percent solids; 20 percent solids content is usual. To transport the mud by mechanical means such as a conveyor belt would require dewatering the mud to at least 55 to 60 percent solids. It does not appear possible by conventional dewatering methods to achieve a mud with this high a solids content except by partial drying. In-plant drying would generally involve substantially higher disposal costs than present mud-lake disposal.* It must be anticipated, therefore, that general practice in the near-term will continue to be impoundment of the mud slurry.

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*One operating company stated that mud-disposal methods now being implemented at that operation will allow the mud to be transported from the plant as material dried to its maximum shrinkage.

(4) The development of a more rapid settling character of a mud has benefits of interest to the operators. Methods used to control the flocculation in the thickener circuit are considered a highly proprietary subject. Use of synthetic flocculants is of continuing interest because synthetics offer opportunities to achieve better quality control of the flocculant and, therefore, better process control. Synthetics have been tested in plant runs with mixed success. Only one processor stated that synthetic flocculants were being used at that site as standard plant practice.

(5) All operators are aware of prior commercial use of red and brown muds of certain size fractions, for example, the making of aggregates for dike construction and as a soil conditioner for acidic soil. However, these have been specialty uses that have involved only a minor amount of the total mud discharged. No major use was reported by the operators.

SECTION V

LITERATURE REVIEW

GENERAL

A detailed review was made of the published technical literature pertaining to the processing of bauxite and the red-mud problem. The period covered by the review was 1940-1975. The following journals were used to make the search:

- Chemical Abstracts
- Engineering Index
- National Technical Information Service (NTIS)
- U.S. Bureau of Mines Reports of Investigations
- Mining Engineering.

Chemical Abstracts was noted to cover Metallurgical Abstracts, Ceramic Abstracts, and World Aluminum Abstracts.

The journals were searched under the following key terms:

- Bauxite refining/treatment
- Red-mud (or brown-mud) dewatering/bauxite refinery waste treatment
- Red-mud (or brown-mud) utilization.

The articles found in the search that were determined by review of the abstracts to be of interest were reviewed in the original and classed in the following categories:

(1) Bauxite Refining
(2) Red-Mud Dewatering
(3) Red-Mud Utilization.

The literature review disclosed more than 600 pertinent articles. Much of the publically available information was found in non-English-language publications. Table 3 shows the number of publications reviewed under each category. Figure 5 shows the overall distribution of articles published in English and non-English languages from the year 1940-1975. After 1950, a majority of articles were found to be in foreign languages. The number of foreign publications on bauxite processing increased sharply in the early 1970's. Approximately 45 percent of the abstracts reviewed were patents. Of these, only 30 percent were in English.

TABLE 3. PUBLICATIONS PERTAINING TO BAUXITE PROCESSING TECHNOLOGY - BY CATEGORY

Title	Number of publications	Language distribution	
		English	Non-English
Bauxite refining	250	78	172
Red-mud dewatering	80	20	60
Red-mud utilization	286	56	230
Total	616	154	462

The literature review established that new methods are being developed to utilize alumina sources other than bauxite. However, the majority of the publications concern modifications of the basic Bayer Process to increase alumina recovery and to reduce alkali (soda) loss.

Developments were reported concerning red-mud dewatering technology. Most of the publications were related to the use of synthetic polymers to flocculate the red-mud suspensions. The use of combination flocculants for example, starches and polymers, metal ions and polymers, and flours and polymers, was described.

The literature search also showed that a substantial amount of research has been conducted on the subject of utilization of red muds. Numerous publications were noted which dealt with recovery of different metals from the muds. Of these, the recovery of iron, aluminum, and titanium shows some potential. Publications were reviewed which concerned use of the red mud in ceramic products. Other publications relate to the application of the red mud in paints, as a fertilizer, and as an adsorbent for gases.

A complete listing of the literature references found in the search is given in Appendix A. Pertinent articles in the various categories were reviewed in detail and the information obtained in each category is described in brief in the following sections.

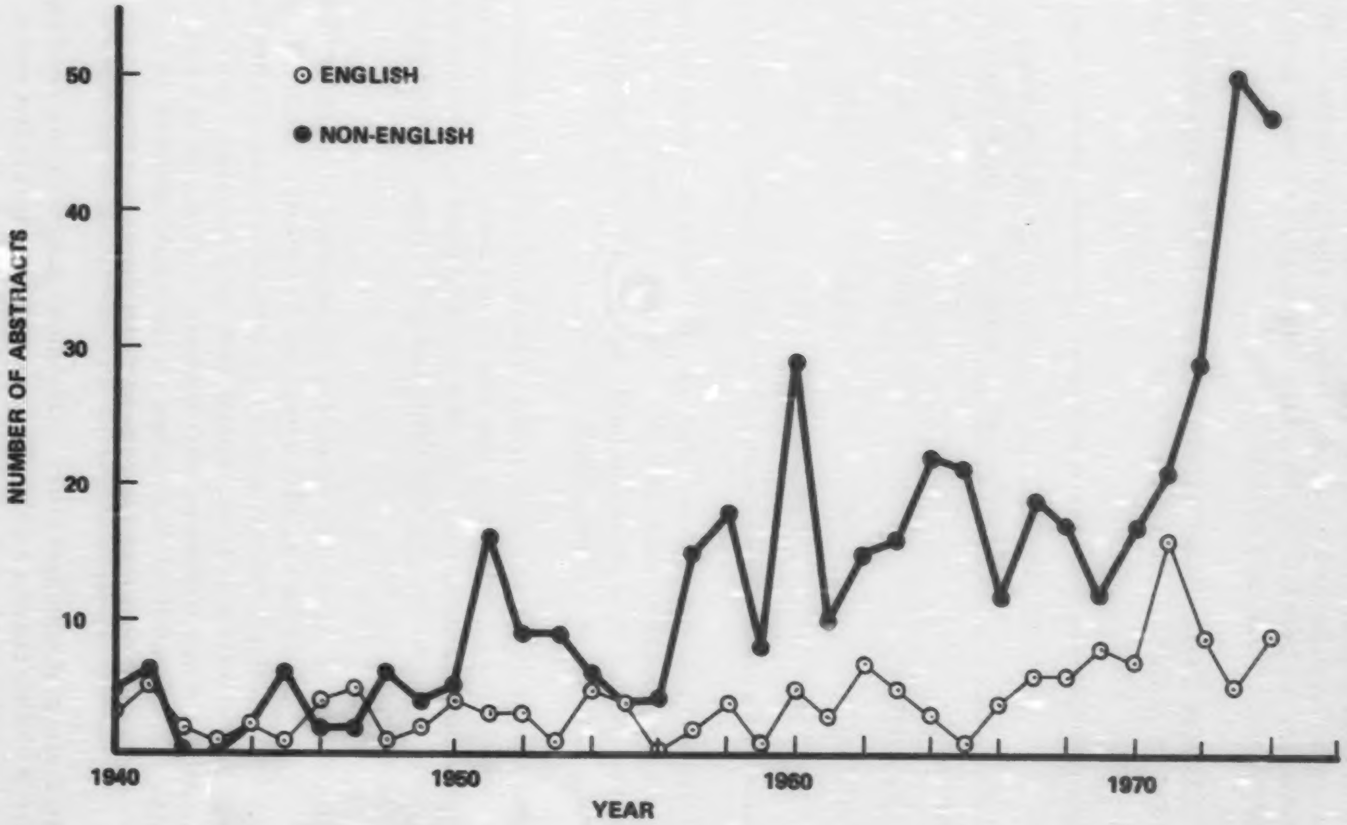

Figure 5. Distribution of literature in English and non-English languages from the year 1940 to 1975.

BAUXITE REFINING

Bayer Process Technology

The Bayer Process is the conventional method used worldwide for extraction of alumina from bauxite. Of the total 250 literature references to refining of bauxite reviewed in this study, approximately 60 percent referred to the basic Bayer Process while the remainder referred to methods to process alternative sources of alumina and various bauxites not suitable for Bayer methods.

In general, hot alkali leaching of the bauxite as is used in the Bayer Process allows recovery of 90 percent or more of the contained Al_2O_3.(5) Various techniques to improve recovery, for example, using additives to the alkali such as Al_2O_3(6), changing the temperature and pressure(7), or grinding the bauxite during digestion in the autoclave(8), are reported, but these offer only a marginal advantage. Dunn(9) recommended use of vertical digesters rather than the more common horizontal units where "sand" or insoluble coarse portions of bauxite are present which tend to be held in suspension in the lower part of the vessel. This was found to avoid scaling of interior of digesters and to decrease retention time needed in the digester. Adjustment of the alumina-to-soda ratio was shown to provide means to improve decantation and also to reduce the amount of soda solution to be concentrated and recycled.(10)

For continuous rather than batch or semibatch digestion of bauxite, a tubular reactor in a turbulent flow condition was proposed by Plass.(11) He recommended heating an ore suspension of 3 to 6 volume percent solids at 95 to 150 C (200 to 300 F) and at 200 atmospheres (3000 psi). Soudan, et al,(12) proposed using a gradually increasing temperature of digestion from 88 to 234 C (190 to 450 F) in two heating tanks and eight autoclaves operated in series. Ninety-five percent solubility of alumina was reported. Similarly, Juhasz, et al,(13) recommended preheating a bauxite-soda mixture in several stages in a shell-tube heat exchanger at 100 to 140 C (212 to 280 F) before digestion in autoclaves.

The use of increased temperature during bauxite digestion was also recommended as a means to increase Al_2O_3 yield and also to allow reduction in the strength of the recirculating solution. This procedure was reported to reduce the heat requirement by 35 to 40 percent.(14, 15, 16) Use of high Na_2O concentration, however, was recommended to avoid Al_2O_3 precipitation in thickeners and to digest excess of bauxite without increasing the digester capacity.(17-19)

The Al_2O_3 recovery was found to be increased by Scholder(20), who proposed decomposing calcined bauxite by heating for several hours at

180 C (360 F) in a liquor containing an alkali metal oxide and a halide such as NaCl, NaBr, or KCl in a ratio of 3:1. Antipin, et al,(21) suggested adding a small quantity of organic matter to leached pulp, while others(22) recommended using a mixture of K-Na alkali mixture to increase Al_2O_3 yield and to decrease alkali loss during the process.

Belyaev, et al,(23) observed improved leaching of bauxite when MgO was used with lime as an activator. Wolf(24) reported that on addition of meta-, tetra-, or perborate to the bauxite at a digestion temperature of 145 to 165 C (290 to 330 F), the Al_2O_3 extraction requires about half the usual time and calcining of the ore may often be omitted. Similarly, addition of lime or soda ash(25) or FeO(26) was noted to reduce the alkali and alumina losses and to improve sedimentation rate of the red mud.

Numerous pretreatment steps and other modifications have been recommended to increase Al_2O_3 recovery in the Bayer Process. Preliminary calcination of bauxite at 260 to 310 C (500 to 600 F) was proposed(27); addition of lime to bauxite and then calcining and grinding of the mixture before digestion was recommended(28); addition of some red mud in digester was reported to be beneficial(29), as was the addition of 0.6 to 0.7 g (1.3 to 1.5 lb) lignin/ton of bauxite(30). One of the proposed methods(31) involved heating of bauxite with NaOH at 220 C (430 F) in a rotating stainless steel autoclave for 10 to 20 minutes and then quenching the apparatus in water to lower the temperature to 100 C (212 F). The resulting suspension when washed with dilute NaOH; the alumina extraction was found to be essentially complete with 0.05 ton NaOH/ton Al_2O_3 consumed.

Gebefuegi(32) reported an improvement in Al_2O_3 recovery by first mixing ground bauxite with 10 percent Na_2CO_3 at 95 to 105 C (203 to 225 F). Then CaO and NaOH was added to the mixture and maintained at 105 to 125 C (225 to 260 F) for 10 minutes before subjecting it to Bayer Process. Other publications on improved methods to increase Al_2O_3 recovery included grinding of bauxite with recycled sodium aluminate solution(33); leaching bauxite with alkaline aluminate solution(34); addition of 5 to 20 g NaCl and/or Na_2SO_4/liter to the digestion system(35); pulverizing and sintering a mixture of bauxite, Na_2CO_3, lime, and coke to make pellets and then grinding it and leaching at 90 C (194 F) with water(36-38); utilizing a tube reactor for bauxite leaching(39); and treating bauxite with CaO and NaCl, which converted goethite to hematite which, in turn, also improved red-mud filterability(40).

Lime-Soda Sinter Process Technology

This process was initially developed by Alcoa during World War II to recover Al_2O_3 from domestic kaolinite. The technique is a modification of the Bayer Process whereby the red mud obtained after the usual Bayer digestion is mixed with soda ash and lime and sintered in a kiln. The sinter product is ground and leached with hot water to extract sodium aluminate liquor which is recycled back in Bayer's circuit.(41) This method is particularly suitable for processing siliceous ores in which the alumina is chemically bound to silica.(42) Flint, et al,(43) described a cyclic process in which high-silica bauxite is boiled with NaOH-NaCl solution, filtered, and Al_2O_3 precipitated with carbon dioxide gas. The leach residue in this process was also sintered with $CaCO_3$, ground, and the Al_2O_3 extracted at 70 to 80 C (160 to 180 F) with water.

Grigoréva, et al,(44) reported that when bauxite was roasted with soda and lime in the presence of solid carbon, less soda was consumed and high Al_2O_3 was extracted. Fusion of bauxite with Na_2CO_3 and $CaCO_3$ gave a porous material which could be leached at low temperature.(45)

Dobos(46) proposed an improved technique involving leaching bauxite by the Bayer Process, then reduction of the red mud by puddling and removal of iron by magnetic separation. The slag was sintered with Na_2CO_3 and CaO and then leached to recover additional Al_2O_3. An alternative to the sinter method proposed by Calhoun, et al,(47) involved calcining bauxite at 980 C (1796 F) and then leaching by percolation with 15 percent NaOH solution at 94 to 96 C (201 to 205 F). This procedure was reported to remove up to 77 percent SiO_2.

Alumina From Low-Grade Bauxite

Numerous methods have been proposed to recover Al_2O_3 from low-grade ores. One method involves reduction to yield a product containing an Al-Fe-Si metallic phase. This can be leached with NaOH to recover sodium aluminate.(48) Digestion of the ore in an autoclave with both NaOH and $Ca(OH)_2$ solution was proposed(49); and use of a tube autoclave with NaOH at 300 to 350 C (570 to 660 F) was recommended(50).

For bauxite which digests slowly in NaOH, the rate and yield can be increased by grinding in a vibratory ball mill which disturbs the internal structure of bauxite and increases the solubility by a factor of six.(51, 52)

Different methods and reagents have been recommended to remove impurities from the leach liquor; for example, HCl and $Ba(OH)_2$ were used to remove organics.(53) Boiling the liquor with 1 percent activated carbon(54) or roasting the bauxite at higher temperature(55-57) are suggested steps. Sulfur, if present, can be removed by agglomerating it with lime and coke and then heating it.(58) Other methods involved treating bauxite with Na_2SO_4 in oxidizing and reducing medium.(59) Phosphorus present in bauxite could be controlled by adding $CaCO_3$.(60)

Desilication of aluminate liquor has been recommended by adding hydrated alkali aluminosilicate(61) or by adding 4 percent white mud (mineral noselite with sulfate)(62) to the heated liquor. General methods recommended to remove most of the impurities referred to removing CO_3 and SO_4 by evaporation and crystallization at 40 C (104 F). On cooling the liquor, a residue rich in P_2O_5 and V_2O_5 was found.(63) Addition of $Ca(OH)_2$ to the liquor was noted to precipitate all salts.(64) Zinc, if present, could be removed by precipitation as ZnS.(65)

Bauxite Beneficiation

Studies on the physical beneficiation of bauxite in general concern the removal of reactive silica. Pickens(66) recommended dispersing the bauxite in an agitated slurry and then removing silica by desliming. Prasad, et al(67) reported that a majority of silica from bauxite could be removed by grinding and screening at minus 20 mesh. Other publications referred to flotation of silica with a cationic collector (e.g., an amine)(68) or, flotation of bauxite with an anionic collector (e.g., oleic acid)(69), gravitation-magnetic separation(70), photoseparator(71), and selective dispersion(72).

Pedersen Process

The process was developed to process ferruginous bauxite containing high SiO_2 and TiO_2 which could not be effectively treated by standard Bayer methods. It involves smelting the bauxite with lime and coke in an electric furnace at 1750 C (3180 F). The iron oxide can be reduced to molten iron with the formation of a calcium aluminate slag; alumina is recovered from the slag by grinding and leaching with Na_2CO_3 solution. The Al_2O_3 is precipitated by neutralizing the liquor with CO_2.(73-75) Some improvement in the leaching step was reported by using a temperature of 80 C (176 F). This allows an increase in the Al_2O_3 content of the solution which then permits precipitation of the Al_2O_3 by seeding.(76)

RED-MUD DEWATERING

The partial dewatering of red mud is an integral part of the Bayer Process operations in the washing circuit for recovery of caustic. Practical considerations require that the mud be thickened to about 20 percent solids. If the degree of thickening is not achieved, plant capacity is adversely affected. For this reason and to reduce the capital cost of thickening and washing, considerable laboratory and in-plant studies have been made of the factors that affect the settling and thickening of the red mud.

Seventy-one literature abstracts pertaining to red-mud dewatering were reviewed. About 85 percent of this literature referred to the flocculation of red mud with a wide variety of reagents. The remainder referred to filtration and centrifugation methods of dewatering. The pertinent literature on dewatering is reviewed in the following paragraphs.

Flocculation

Flocculation is the addition of a chemical agent which causes the fine particles in suspension to agglomerate and, therefore, settle more rapidly by gravitational force. The common flocculating agent used in the alumina extraction industry is cooked starch. It was reported that the method of preparation of the colloidal starch solution was important; for example, heating the starch solution makes it a more effective flocculation agent.(77) Some investigators(78, 79) reported starch phosphate as being more effective than natural starch. The use of rye bran, rye flour, and corn flour as a red-mud flocculant has also been tried.(80, 81) The most effective of these were found to be rye bran and corn flour in the range of concentration from 0.1 to 0.125 g/l of slime pulp.

Numerous publications describe the use of synthetic polymers (polyacrylamide) as a red-mud flocculant. Wolf, et al,(82) reported different sedimentation rates and end volume percent for red mud, depending on the molecular weight of the polymer used. In general, the high-molecular-weight polymers are more effective than the lower molecular weight ones. Generally, also, a lesser amount of polymer [50 to 100 g/ton (0.11 to 0.22 lb/ton) of red mud] is required compared to the amount of natural starch needed [1 kg/ton (2.2 lb/ton of red mud].(83, 84) Sullivan(85) reported an increase in settling rate of red-mud slime by adding 0.0125 to 0.2 weight percent solution of an ultrahigh-molecular-weight polyvinyl aromatic sulfonate and also by the addition of lignosulfate product (about 25 weight percent active lignosulfonic acid). Polysodium acrylate prepared by irradiation was also reported to be an effective coagulant for red mud.(86) Ismatov, et al,(87) reported an optimum settling of solids at a polymer concentration of $\leq$125 g/ton and liquid:solid ratio of 20:1. Werner(88) reported that the polysodium methacrylate gave the highest settling rate of all the polymers investigated. A higher sedimentation rate was also reported when an emulsion of polyacrylic acid ester and polymethylacrylate was used.(89)

Skobeev(90) reported a threefold increase in settling rate of red-mud slurry using a flocculating mixture of 6.25 g/m^3 polyacrylamide and 25 g/m^3 flour. Sibert(91, 92) observed that the separation rate of dispersed red mud was increased when treated with cooked starch and a polymer of molecular weight greater than 50,000.

Addition of certain minerals was shown to accelerate or retard settling of red mud, e.g., rutile, magnetic, melanterite, and siderite accelerated the settling; limonite, kaolinite, quartz (opal), and pyrite retarded the settling rate of the red-mud suspension.(93-95) Chu, et al,(96) reported that the addition of CaO to the Bayer Process increased the extraction of Al_2O_3 and increased the sedimentation rate of the red mud. He also reported that addition of lime and BaO increased, while MgO retarded, the sedimentation rate of red-mud suspension.

Makary, et al,(97) reported using homogenizing wheat flour with caustic for red-mud sedimentation, while Goldman, et al,(98) obtained positive results with a composite flocculant consisting of 1.1 ratio of polyacrylamide and rye flour.

A mixture of 0.1 to 2.0 weight percent starch (based on red mud) and 0.3 to 12 times the weight of starch of calcium compounds, e.g., $Ca(OH)_2$, $CaCO_3$, and CaO, gave a high sedimentation rate compared to that obtained by starch alone.(99) Similarly, a mixture of calcium compounds and a polymer, lime Na polyacrylate, accelerates sedimentation and filtration of red mud.(100)

Derevyankin, et al,(101) reported that an optimum concentration of Na_2O (15 to 20 g/dm^3) is necessary for attaining maximum settling rate. Other publications on red-mud sedimentation dealt with addition of sea weeds(102), clarified aluminate solution(103), gelatinizing(104), freezing and thawing of red mud(105), and use of higher temperature(106).

Centrifugal Dewatering

Centrifugal methods studied for partial dewatering of red muds included the use of the hydroclones and centrifuges. However, the number of studies done were limited. German-Galikana, et al,(107) found that hydroclones installed ahead of thickeners are able to eliminate 75 percent of solids provided that the liquid-to-solid ratio in the cyclone feed is maintained at 0.8 to 1.0.

Good, et al,(108) showed an increase in solids content of sludge from 20 to 25 percent to 40 percent with a centrifuge. They also reported an increase in centrifuge capacity by about 35 percent by addition of a flocculant.

Filtration

Polyakov, et al,(109) concluded that for efficient filtration of the red mud, the liquid-to-solid ratio should be 1.8:1, and that the rate of filtration increases proportionally to the increase in pulp temperature and inversely to total thickness of cake layer. These workers also reported that filtration rate depends little on the Na_2O or sodium aluminate contents in the pulp within 5 to 40 g/l (0.011 to 0.088 lb/ton) concentration.

Kaempf(110) and Plaetschke(111) have reported that rotary drum filters are more economical than the conventional Kelly press for red-mud filtration due to reduced labor and wash water requirements. The Na_2O losses were also noted to be reduced considerably. Konig(112) reported achieving a filtration rate of 125 to 155 kg/m^2 (25 to 32 lb/ft^2) with a rotary drum filter. Recently, Vogt(4) reported a filtration rate of 190 kg/m^2 (39 lb/ft^2) of Jamaican red mud using a rotary drum filter operating at 75 C (167 F). The filter cake obtained had 40 percent solids and was thixotropic in behavior.

Martine(113) reported that red mud obtained from previous filtration is more effective as a filter aid than the conventional materials such as diatomite.

UTILIZATION OF RED MUD

The search of the literature pertaining to possible commercial use of red mud was organized in three categories: (1) metallurgical, (2) ceramic, and (3) waste treatment. The references totaled 267 articles, and more than half concerned metal recovery from the mud. Ceramic-use references totaled 96 articles, or approximately 30 percent of the citations. The pattern that the recent research has been done largely outside the United States is clearly established. More than 90 percent of all literature in the field of red-mud utilization was based on the work of foreign investigators.

Metallurgical

Literature references on metal recovery totaled 140 articles classed as shown in Table 4. It can be noted that approximately half the references pertain to recovery of iron. Recovery of titania and additional alumina is also proposed. The higher value metals such as niobium, gallium, and vanadium have received attention but are present in such low concentration that their commercial recovery has not been tried.

TABLE 4. CLASSIFICATION OF LITERATURE BY SUBJECT ON METALLURGICAL USES FOR RED MUD

Material recovered	Number of articles
Alumina	12
Titania	11
Iron	62
Iron, alumina	20
Iron, titania	2
Miscellaneous metals	33
Total	140

A listing of these references by country of origin is given in Table 5.

TABLE 5. CLASSIFICATION OF LITERATURE ON RED-MUD UTILIZATION BY COUNTRY (METALLURGICAL)

Country	Number of abstracts
U.S.S.R.	37
Japan	18
Hungary	24
U.S.A.	9
Germany	10
France	10
Czechoslovakia	6
India	4
Yugoslavia	3
England	1
Italy	1
Canada	1
Spain	1
Belgium	1
Korea	1
Unidentified	13
Total	140

Iron—Several processes have been developed to recover iron from the red-mud residues. One method is the carbon-lime-soda sinter process which can be applied either to ore or to the red mud.(114, 115) In this process, the iron is reduced and recovered by magnetic separation from the waste residues after alumina leaching. A U. S. patent has been issued describing the application of a fluidized bed to produce sponge iron by these techniques.(116) Another patent was granted for a process to treat high-iron-content bauxite ores involving reductive roasting with magnetic separation of iron from the leach residues.(117)

Direct electric arc smelting of the red mud has been proposed for recovery of iron from high-iron-content bauxites. In this case, pig iron can be produced with up to 98 percent recovery of iron value in the bauxite. The slag from the smelting operation can also be further treated to recover up to 84 percent of the alumina lost by the Bayer Process. This particular process was recently advocated by the McDowell Wellman Engineering Company as being both technically and economically feasible and they have developed the process through a pilot-scale stage.(118) The economics assume that the pig iron or steel would be produced near the bauxite refining plant to take advantage of low-cost iron units in the red mud. The U. S. iron and steel industry has not taken steps to commercialize the process. Economics require a low-cost means to completely dewater the mud. Trace elements, for example, phosphorus, can have a very significant and adverse effect on steel quality.

Alumina and titania recovery - Alumina and titania recovery from the red mud are only of secondary interest. However, if the mud is smelted for iron recovery, the slag from the smelting operation can be leached with sodium carbonate solution to recover most of the alumina values. Titania can be recovered by leaching the residue of the carbonate leach with sulfuric acid.(114) The recovery of titanium from the red mud is technically feasible but the complicated processing is too costly to compete with the recovery from natural titanium ores such as ilmenite or rutile.

Other metals - Various other rare metals such as gallium, vanadium, and scandium can be recovered from the red-mud residues or at various stages in the Bayer Process. It has been reported that gallium recovery is economical by direct electrolysis of the caustic aluminate liquors.(119) Several studies have also been conducted on vanadium recovery. In one method a vanadium slag is separated from the pig iron mud.(120). In another method, liquid-liquid extraction by amines is used on the leach liquors from the Bayer Process to recover vanadium.(121)

Again, it appears that these recovery methods are only of secondary interest after first extracting the iron values from the red-mud residues.

Ceramic

For most of the possible uses of the mud in ceramics, it would be necessary to dry or at least partially dry the mud, for example, before firing in a brick kiln. Certainly the red mud would require drying to allow transportation for distances greater than 50 miles. Drying would likely entail costs of the order of the present cost of most raw materials for the large-tonnage ceramic products. Although certain specialty uses might be made of the mud, they are of little significance, since uses relate to the overall problem of utilization of the muds to eliminate the disposal problem. Therefore, review of the literature was done using the general criteria that uses offering the greatest potential would be those that are large-volume applications, require minimum pretreatment of the red mud, and require minimum transportation, e. g., less than a distance of 50 miles.

Cement, building blocks, or brick and to a lesser extent lightweight aggregate and rubber are potential large-volume applications where red mud might be used. It is expected that a minimum pretreatment (dewatering) would be required for use of red mud in brick and lightweight aggregate. For use in cement and as a rubber filler, acid washing would be required and complete drying and powdering would be necessary for the filler application. Cement and brick manufacturing plants are fairly widely distributed throughout the United States, with lightweight aggregate plants being less numerous. There is potential for such plants being located near the source of the red mud which is considered necessary to minimize transportation costs.

Other applications for red mud, such as those listed in Table 6, as well as applications in exothermic mixes, as a scouring or polishing medium, or as a drilling mud, are considered of low potential for either technical, economic, or low-volume reasons.

Ninety-six papers were found on uses of red mud in ceramic applications. The various uses are shown in Table 6. Table 7 indicates the degree of activity, by country, relative to developing uses for red mud.

From these tables, it can be noted that about 65 percent of the abstracts dealt with applications of red mud as cement, construction block material, lightweight aggregate material, plastic and resin filler, and pigment. The other 35 percent of the abstracts concerned 13 other applications.

Five countries, Japan, Germany, France, the U.S.S.R., and Hungary, were the origin of slightly more than 75 percent of the publications. Three abstracts were of U.S. origin.

Cement – Three potential applications for red mud as a cement material, with varying degrees of promise, were indicated in the literature.

Cement raw material – Portland cement generally consists principally of silicates and aluminates as shown in Table 8.

Thus, the oxides of calcium, aluminum, silicon, and to a minor extent iron make up the major portion of the cement. A typical red mud contains CaO, SiO_2, and Fe_2O_3 in the range around 5 to 10 percent, 2 to 10 percent, and 40 to 50 percent, respectively. Thus, its potential use as a raw material for cement manufacture has been of interest.(112, 123) However, the amount of red mud that might be incorporated directly as a raw material would be low because it contains a relatively high iron oxide.(124)

Additions of ~5 percent of treated red muds to portland cement were reported to increase strength and affect setting time.(125-128) Greater additions decreased strength. Pretreatments of the red-mud slurry included carbonic or sulfurous acid treatments, drying or calcining, and grinding. Untreated red-mud additions in small amounts had some affect on setting time.

TABLE 6. CLASSIFICATION OF LITERATURE BY SUBJECT ON CERAMIC USES FOR RED MUDS

Field of application	Number of abstracts
Cement material	22
Construction block material	16
Lightweight aggregate material	10
Plastic and resin filler	9
Pigment	6
Miscellaneous materials recovery	6
Caustic recovery	5
Catalyst material	4
Fertilizer material	4
Coating material	3
Insecticide material	2
Refractory cement material	2
Road, pavement, soil stabilization material	2
Metal surface treatment material	1
Sewage treatment material	1
Glass material	1
Insulation material	1
Coke additive	1
Total	96

TABLE 7. CLASSIFICATION OF LITERATURE ON RED-MUD UTILIZATION BY COUNTRY (CERAMIC)

Country	Number of abstracts
Japan	32
Germany	21
France	8
USSR	9
Hungary	6
U.S.	3
India	2
England	2
Taiwan	2
Czechoslovakia	2
Australia	1
Switzerland	1
Ukraine	1
Poland	1
Unidentified	5
Total	96

TABLE 8. RAW MATERIALS IN PORTLAND CEMENT

Component	Amount, percent
Tricalcium silicate	45
Dicalcium silicate	27
Tricalcium aluminate	11
Tetracalcium aluminum ferrite	8
MgO	3
Other miscellaneous	6

Cement for iron ore briquetting - Cements produced by sintering red mud or red mud and limestone have been considered as possible binders in iron ore briquetting.(129, 130) For this application, the relatively high Fe_2O_3 content of the red mud is an advantage.

Construction block and brick material - The use of red mud for making structural shapes was considered in 16 publications. However, it was found that the resulting products were mechanically weak. The compressive strength of the standard U.S. common bricks exceeds 8000 psi. In most cases the red-mud products had strengths less than this. This was true even in the case of brick made of mixtures of red mud and clay fired at temperatures comparable to those used in firing all-clay brick.(131, 132) A 5 to 10 percent addition of red mud to clay was used to make roofing tile.(133)

However, brickmaking could be an important possible use of the muds. Construction, and thus the structural brick industry, is currently below its peak of several years ago. However, in 1973, around 350 plants in the U.S. were producing 18 million tons of brick annually. Sixty-four of these plants were in Alabama, Arkansas, Louisiana, and Texas. At a 300 ton/day typical capacity of a plant, these 64 plants would account for 19,200 tons of brick/day.

It is judged from the literature that it is not likely that the red-mud content of brick can exceed 50 percent and that 25 percent is a more likely prospect. Brick with more than 50 percent red mud were mechanically weak by U.S. standards.

If the 64 plants cited above made brick comprising 25 percent red mud, the daily red-mud consumption (on a dry red-mud basis) would be 4,800 tons. It is questionalbe that all of the 64 plants could use red mud because of the distance of some of them from the source of the red mud, resulting in prohibitive shipping costs. If one-fourth of these 64 plants could use the red mud, 1,200 tons/day could be consumed. This is considered a likely maximum use of red mud for brickmaking but is still a significant use.

Possible contributions of a red-mud addition to clay in brickmaking include

- Pleasing color and color shades dependent on content
- Improved dry strength for handling before firing
- Lower firing temperature and thus fuel or energy savings.

In the evaluation of this use of red mud, it is recommended that various red muds be blended with a light (color) burning clay in various ratios. Specimens formed from this mixture would be evaluated for dry strength,

strength after firing at various temperatures, water absorption, and color. The water absorption, as well as fired strength, would indicate whether the typical 1900 F firing temperature was appropriate or whether a lower temperature might be tolerated or even preferred.

Lightweight aggregate material - Lightweight aggregate is used with cement to make a lightweight strong concrete. Some clays or shales bloat and expand when heated to give the lightweight aggregate. Others do not. The literature(134-138) indicated that combinations of red mud and non-bloating clays or shales would bloat on heating to yield lightweight aggregate. One mechanism accounting(136) for the bloating is the evolution of CO_2 gas resulting when Fe_2O_3 in the red mud converts to Fe_3O_4 on heating. The oxygen that is evolved during heating of the mud combines with carbon from organic matter to give the CO_2. Red mud:clay ratios for making lightweight aggregate in the range of 50:50 to 90:10 were given which could potentially represent a significant use of red mud in this product.

It is estimated that the lightweight aggregate industry is about one-eighth the size of the brick industry. However, it also appears from the literature that a lightweight aggregate comprising 75 red mud (dry basis) - 25 clay or shale is feasible. This would be three times the maximum consumption in brick using 25 red mud - 75 clay. Thus, at a maximum daily consumption of 1200 tons of red mud/day for brickmaking, the maximum consumption for lightweight aggregate production would be of the order of

$$\frac{1200 \text{ tons} \times 3}{8} = 450 \text{ tons/day.}$$

A possible attraction to the use of red mud in lightweight aggregate lies in location of the plant site. Since red mud can be the predominant raw material, possibly up to 90 percent in content, location of the plant should be at or near the source of the red mud. The clay or shale, which apparently need not possess bloating characteristics itself and which comprises the minor raw material, can then be shipped to the red-mud source.

There could be merit in study of the mixing of red muds with nonbloating clay or shale in the ratios of the order of 75 red mud - 25 clay and the mixes granulated and fired to determine bloating characteristics. Evaluation of the fired granulated material would be by visual inspection and bulk density measurement, the latter to determine how "lightweight" the aggregate is.

Plastic and resin filler - Though numerous materials can be used as fillers, it was considered significant that red mud as a filler in rubber gave a rupture strength higher than all other fillers excepting carbon black.(139-141) It should thus be a suitable substitute for carbon black in some applications. Thus use as a filler requires that the red-mud slurry be acid washed, dried, and powdered.

Pigment - Red mud was indicated to have possible application as a pigment for coloring concrete(142), glass(143), and paint(144). Consistency of color is a requirement for any pigment application. This factor could make it difficult to use the mud for this purpose.

Miscellaneous applications - There was no evidence that red mud has unique yet useful characteristics not to be found in other materials. Basically, it is a "filler" type of material which can be used in some applications. For instance, it can be used as an insecticide carrier(145, 146), a soil liming equivalent(147), a coating filler(148, 149), a soil stabilization material(150), or a fertilizer(151, 152) if potassium or nitrogen chemicals are added to it. Other materials can serve these same purposes.

Some of the publications(153, 154) dealt with the recovery of caustic associated with the red mud while others mentioned its application as a catalyst material(155, 156), a refractory cement material(157), a metal surface treatment ingredient(158), a sewage treatment material(159), an insulation material(160), and a coke additive(161).

Waste Treatment

Red mud has been tested as a possible agent in a number of waste-treatment-abatement applications involving both gaseous and liquid effluents. For example, it has been found effective as an adsorbent for various chemical pollutants such as sulfur oxides(162, 163) and H_2S(164) from gaseous effluents. It has also been reported as a good adsorbent of arsenic(165) from industrial wastewaters. In addition to these aspects, it has been investigated as a coagulant or flocculating agent(159, 166) for municipal wastewater treatment.

The total number of articles published on different waste-treatment applications and by country of origin are listed in Tables 9 and 10.

TABLE 9. CLASSIFICATION OF LITERATURE ON WASTE-TREATMENT APPLICATIONS OF RED MUD

Application	Number of articles
Adsorption of sulfur oxides	14
As a coagulant	8
Adsorption of H_2S	7
Others	2
Total	31

TABLE 10. CLASSIFICATION OF LITERATURE ON WASTE TREATMENT APPLICATIONS OF RED MUD BY COUNTRY

Country	Number of articles
U.S.S.R.	4
Japan	11
U.S.A.	7
Germany	4
France	1
England	1
Unidentified	3
Total	31

Adsorbent for sulfur oxides – Dried or calcined red mud has been found effective for adsorption of sulfur dioxide from industrial waste gases.(164) In this investigation, the gases were passed through an adsorption bed of red-mud granules at modest temperatures. The adsorbent bed was found to be over 90 percent effective in removing SO_2 and could be regenerated with a reducing gas such as hydrogen. Similarly, good results were obtained when red mud was mixed with coal for SO_2 control in industrial boilers.(162)

Use as a coagulant – An interesting possible use of red mud is as a coagulant for the treatment and sedimentation of municipal wastewaters and for sludge flocculation. This has been done on a commercial scale in England before the war.(159) The process involves treatment of the red mud with concentrated acids, then drying at modest temperatures, and crushing to give a granular product. Either sulfuric or hydrochloric acids can be used to convert the iron and aluminum oxides in the red mud to sulfates or chlorides. Thus, ferric chloride or alum can be produced which are well known for their coagulating properties. In addition to these effects, it has also been reported that the red-mud materials are effective for removing phosphorus from the municipal wastewaters with 70 to 80 percent effectiveness.

SECTION VI

EXPERIMENTAL PROGRAM

There are substantial differences in various bauxites and the red muds or residues from current alumina recovery operations. A part of this study concerned acquisition of samples and a comparison of the chemical and physical characteristics of these materials.

In addition to analytical characterization, it was considered important to examine the relative ability to dewater the muds. Particular attention was given to factors affecting the flocculation and settling of mud suspensions using various synthetic polymer flocculants. The influence of dissolved metals on the coagulation of the muds was studied. Limited experiments were made on mechanical dewatering, magnetic flocculation, and flotation as possible methods to achieve partial dewatering. These experiments were intended to provide a basis to judge the practical limit in partial dewatering by methods conventionally applied in general processing of minerals.

In addition to the analyses and characterization of the bauxites and mud samples, several experiments were done to verify results previously reported in the literature concerning a kiln pretreatment of bauxite before leaching that might allow magnetic separation of iron from the residue after leaching for recovery of alumina. The results of these experiments are provided in this section of the report.

SAMPLE ACQUISITION

Mud samples were provided to Battelle from five alumina refineries. These samples were obtained as slurry samples, generally from the pipeline going to the mud lake. The slurry samples were sent to Battelle in 55-gallon drums.

The samples were prepared for study by first mixing thoroughly, using a motor-driven agitator to mix the entire sample. When it was determined that all settled material was in active suspension, 5-gallon aliquot samples were taken from the drum for the various tests and analyses. *

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*The brown-mud sample provided by Reynolds was not effectively redispersed after receipt by Battelle. This mud appeared to have partially set with cementlike character.

A listing of the samples received along with the initial characterization is provided in Table 11. The solids pulp density provided in the table was measured by weighing 200 ml of the well-mixed pulp, and then filtering and drying the solids at 105 C for subsequent weighing.

TABLE 11. INITIAL CHARACTERIZATION OF MUD SAMPLES

			Physical properties of mud slurry			
Company	Location	Type bauxite	Color	pH	Percent solids	Pulp density
Kaiser Aluminum	Gramercy, Louisiana	Jamaican	Red	11.7	22.1	1.16
Alcoa	Mobile, Alabama	Surinam-African	Reddish-orange	11.9	7.8	1.08
Alcoa	Point Comfort, Texas	Surinam-Caribbean-Australian-African	Red	11.8	31.0	1.30
Reynolds Metal	Hurricane Creek, Arkansas	Arkansas	Brown	11.7	34.5	1.33
Martin-Marietta	St. Croix, Virgin Islands	Boké (Africa)	Red	12.5	35.6	1.27

CHARACTERIZATION OF MUD SAMPLES

The mud samples as received at Battelle were analyzed for chemical composition, particle size distribution, specific gravity, and settling rate. It should be noted that these samples may be typical but not necessarily representative of the average mud compositions. Details of these analyses studies are presented below.

Composition

The wet chemical analysis of the dried muds and the supernatant liquid associated with the as-received samples is given in Table 12.

The results of semiquantitative analyses by optical emission spectroscopy (OES) are given in Table 13.

Particle Size Distribution

The particle size distribution of the mud samples was determined by wet screen analysis in the size above 44 microns and by Coulter counter

TABLE 12. CHEMICAL ANALYSES OF MUD SAMPLES
(PERCENT BY WEIGHT)

Component	Kaiser Aluminum, Gramercy, Louisiana, Jamaican bauxite		Alcoa, Mobile, Alabama, Surinam-African bauxite		Alcoa, Point Comfort, Texas, mixture of four bauxites		Reynolds Metal, Hurricane Creek, Arkansas, Arkansas bauxite		Martin-Marietta, St. Croix, Virgin Island, mixture of bauxites	
	Dry mud	Supernatant liquid[(a)]	Dry mud	Supernatant liquid[(a)]	Dry mud	Supernatant liquid[(a)]	Dry mud	Supernatant liquid[(a)]	Dry mud	Supernatant liquid[(a)]
Al_2O_3	15.00	0.58	29.10	8.79	17.80	3.34	6.31	1.89	33.80	N.A.
Fe_2O_3	51.50		28.20		40.00		10.70		22.90	
SiO_2	1.70		10.80		9.59		19.90		8.49	
TiO_2	6.70		12.70		8.48		3.32		12.90	
CaO	7.0		5.72		7.57		42.50		3.52	
Na_2O	0.97	1.11	1.34	15.96	2.69	14.24	1.34	7.62	6.0	N.A.
LOI[(b)]	9.30		12.90		10.30		5.04		12.4	

(a) The amounts are reported in grams/liter.

(b) Loss on ignition at 1000 C.

TABLE 13. OPTICAL EMISSION SPECTROGRAPHIC ANALYSES OF MUD SAMPLES

(Percent by Weight)

Element	Kaiser Aluminum (Jamaican)	Alcoa, Mobile, Alabama (Surinam-African)	Alcoa, Point Comfort, Texas (Surinam-Australian-African)	Reynolds Metals Hurricane Creek, Arkansas (Arkansas)
Al	2-4	5-10	3-6	1-3
B	<0.005	0.005	0.005	0.005
Ba	0.02	0.01	0.01	0.01
Be	<0.0001	<0.0001	<0.001	<0.001
Ca	5-10	3-6	4-6	20-40
Co	0.01	<0.005	0.01	<0.002
Cu	0.02	<0.005	0.01	0.002
Cr	0.1	0.05	0.1	0.005
Fe	10-20	5-10	20-40	5-10
K	0.03	0.2	0.1	0.3
Mg	0.1	0.03	0.1	0.3
Mn	1.0	0.02	0.4	0.2
Na	0.5	1-3	2-4	1.0
Ni	0.1	<0.005	0.03	0.002
Pb	0.02	0.01	0.02	0.005
Si	0.8	2-4	2-4	5-10
Sr	0.05	0.01	0.03	0.03
Ti	2-4	3-6	2-4	1-2
V	0.1	0.1	0.03	0.01
Zr	0.1	0.2	0.1	0.2

technique for particles less than 44 microns in size. For Coulter counter studies, the particles were suspended in the supernatant liquid and were thoroughly dispersed with an ultrasonic bath. These data are given in Table 14 and are shown as composite curves in Figure 6. Particle-size analyses were also performed on the Gramercy and Point Comfort mud by the Andreason pipette technique for particle sizes finer than 44 microns. The results for the Alcoa - Point Comfort, Texas, mud are presented in Figure 7 for both the techniques. The results are in general agreement, with each method giving about 50 percent of the mud less than 4 microns.

Specific Gravity

Specific gravities of the dried mud samples were determined with a pycnometer. The specific gravities were calculated as follows:

$$\text{Specific Gravity} = (C-A)/[(B-A) - (D-C)] \quad ,$$

where: A = weight of pycnometer (plus stopper), B = weight of pycnometer filled with water, C = weight of pycnometer and some solids, and D = weight of pycnomer plus solid plus water. The results obtained are given in Table 15.

DEWATERING STUDIES

Dewatering of suspended solids as it applies to this discussion refers to partial dewatering by removal of bulk and interstitial water from the suspension. Such physical dewatering methods as sedimentation, filtration, and centrifugation are of interest. Coagulation and flocculation induced by addition of chemical agents to the suspension can agglomerate the solids into larger and more compact aggregates which in turn are more readily dewatered. Therefore, the influence of such agents is pertinent to this study.

The mud samples used for the dewatering studies were obtained from the pipeline going to the mud lake. Hence, the muds were in a partially flocculated state. Dewatering studies presented in this report do not simulate the plant flocculation of mud, but pertain to dewatering of mud being discharged to the lake.

Sedimentation Experiments

The partial dewatering of suspended solids by sedimentation is induced by gravity and follows the generalized pattern illustrated in Figure 8. A plot of the height or volume of the settled solids versus settling time provides initial and final settling rates calculated from the slopes of

TABLE 14. PARTICLE-SIZE-DISTRIBUTION DATA[(a)] FOR VARIOUS MUDS

Size	Kaiser Aluminum Company, Gramercy, Louisiana, Jamaican Bauxite		Alcoa, Mobile, Alabama Surinam-African Bauxite		Reynolds Metals, Hurricane Creek, Arkansas, Arkansas Bauxite		Point Comfort, Texas Jamaican-Australian-African Bauxite	
	Weight percent	Cumulative weight percent	Weight percent	Cumulative weight percent	Weight percent	Cumulative weight percent	Weight percent	Cumulative weight percent
+28 mesh	--	--	0.2	0.2	--	--	0.1	0.1
28 x 48	0.1	0.1	1.7	1.9	0.4	0.4	0.4	0.5
48 x 100	0.4	0.5	6.8	8.7	8.5	8.9	1.0	1.5
100 x 200	1.2	1.7	11.8	20.5	19.7	28.6	2.9	4.4
200 x 270	0.6	2.3	3.1	23.6	6.7	35.3	1.3	5.7
270 x 325	0.4	2.7	1.5	25.1	4.7	40.0	0.7	6.4
325 x 35 microns	0.8	3.5	0.9	26.0	1.0	41.0	--	--
Average Diameter, microns								
29	0.2	3.7	0.9	26.9	1.1	42.1	0.7	7.1
13	0.8	4.5	1.6	28.5	3.7	45.8	0.7	7.8
18	1.3	5.8	1.6	30.1	4.3	50.1	0.8	8.6
15	1.8	7.6	1.5	31.6	4.3	54.4	1.3	9.9
12	1.8	9.4	1.8	33.4	3.7	58.1	1.5	11.4
9	2.2	11.6	1.5	34.9	3.4	61.5	3.6	15.0
7	3.9	15.5	3.4	38.3	3.8	65.3	5.9	20.9
6	7.0	22.5	10.4	48.7	4.7	70.0	8.6	29.5
5	19.0	41.5	15.6	64.3	7.1	77.1	13.9	43.4
4	14.8	56.3	7.6	71.9	5.8	82.9	16.3	59.7
3	5.7	62.0	4.8	76.7	9.5	92.4	9.0	68.7
2	13.1	75.1	8.4	85.1	4.2	96.6	8.2	76.9
1.5	19.1	94.2	9.9	95.0	2.0	98.6	9.7	86.6
1.2	4.8	99.0	4.4	99.4	1.0	99.6	8.6	95.2
0.9	1.0	100.0	0.6	100.0	0.4	100.0	4.8	100.0

(a) Obtained with Coulter-counter method.

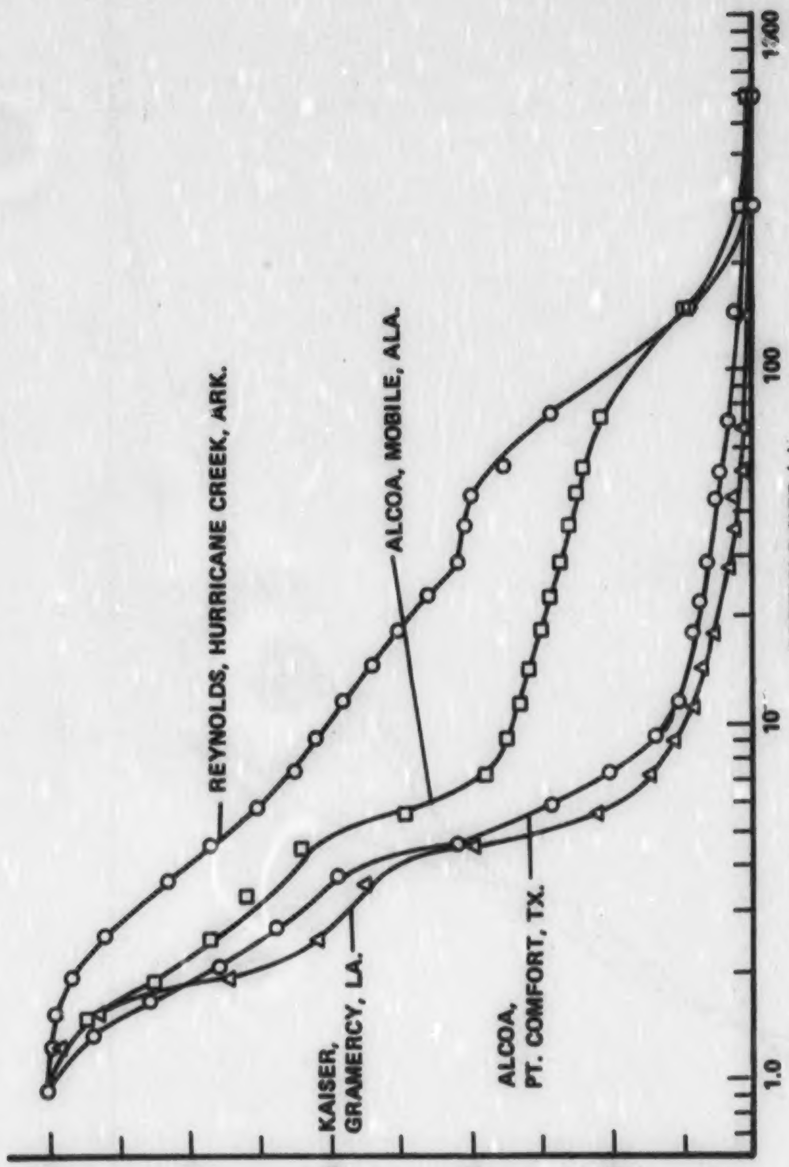

Figure 6. Particle-size distribution of various muds

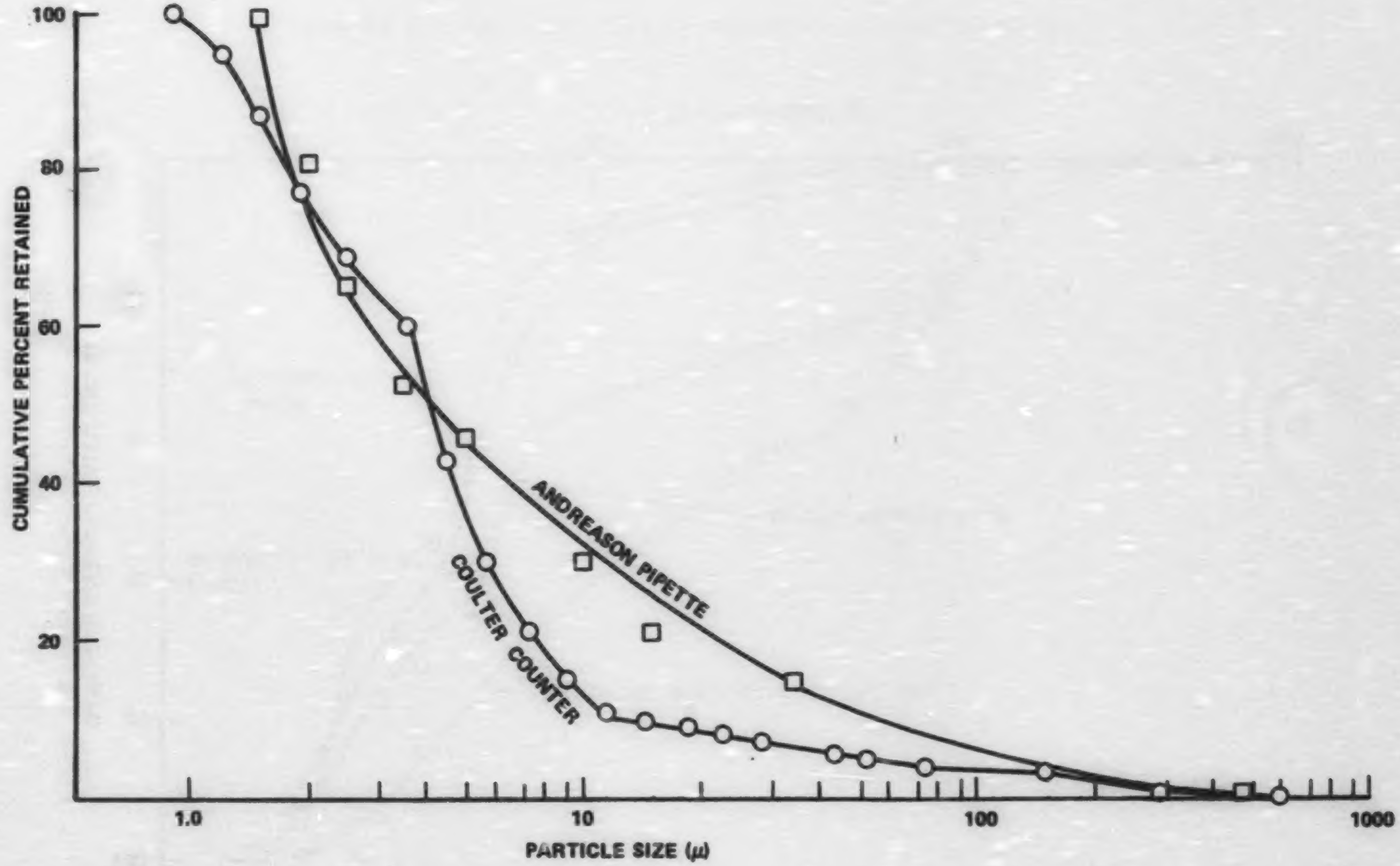

Figure 7. Particle size distribution of Alcoa-Point Comfort, Texas, red mud using Coulter-Counter and Andreason pipette methods

TABLE 15. SPECIFIC GRAVITY OF MUD SAMPLES

Company	Mud Type	Specific Gravity
Kaiser	Jamaican Red Mud	3.10
Alcoa	Surinam - African Red Mud	2.67
Alcoa	Australian - African - Jamaican - Surinam Red Mud	2.69
Reynolds	Arkansas - Brown Mud	2.65
Martin-Marietta	Boké (South Africa) - Red Mud	2.89

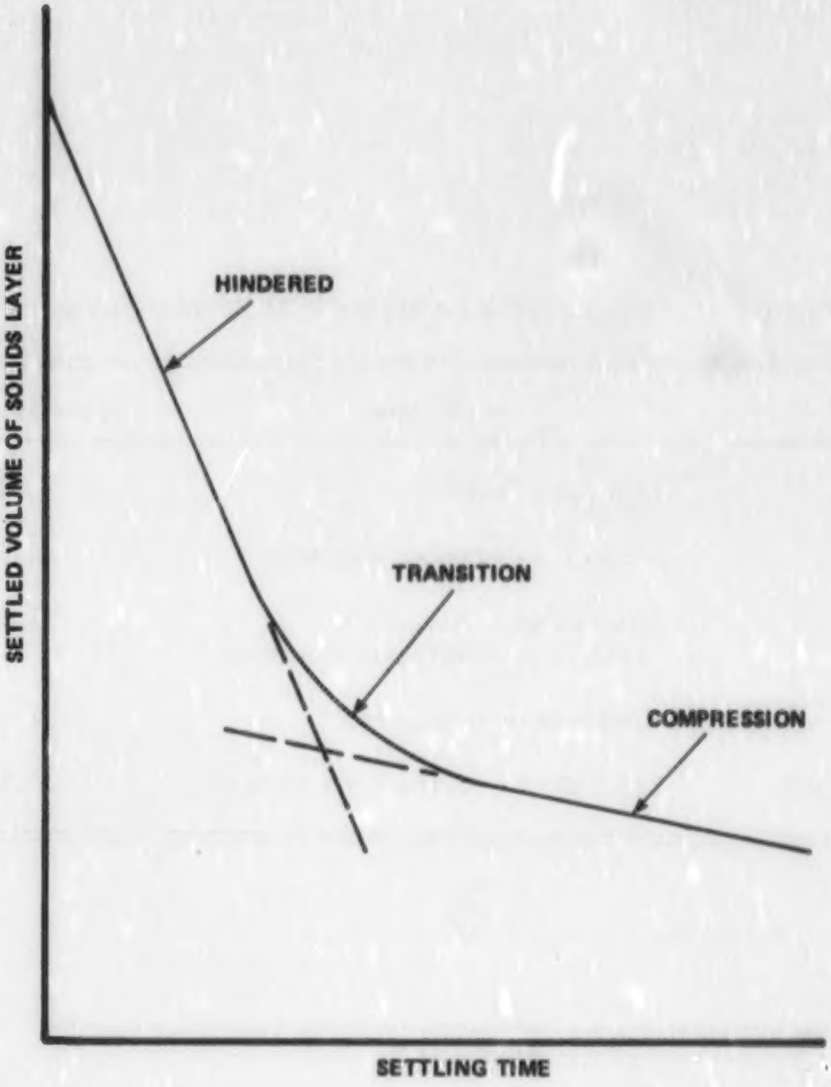

Figure 8. Generalized settling rate curve

the straight line representing the hindered and compression regimes, respectively, as shown in the illustration.

The settling-rate studies of the red-mud-samples suspensions were performed in graduated cylinders. The mud slurry, after gentle but thorough mixing, was poured into the cylinder and the settling tests were begun. The volume corresponding to the heights of the developing interfaces between settled solids and bulk overhead liquid were measured at intervals during a total period of up to 24 hours. The final volume of the solids layer was noted and the percent solids in the settled sludge was determined by weighing the sludge before and after filtering and drying. The sedimentation rates reported are based on measured rates in the hindered settling regime. The initial part of the experimental study involved sedimentation experiments without any addition of chemical reagents to establish the differences in the individual samples received. Subsequent work was done to examine the influence of various flocculants and solution chemistry on the sedimentation.

The settling-rate data for the as-received mud samples are plotted in Figure 9. It was noted that the Kaiser-Gramercy (Jamaican) mud, the Alcoa-Point Comfort mud, and Martin-Marietta-St. Croix mud* had a very similar slow settling behavior. The Alcoa-Mobile (Surinam-African) mud and the Reynolds-Hurricane Creek (Arkansas) brown-mud sample showed substantially higher initial settling rates. With the exception of the Arkansas brown mud, the final settled density of the red muds was 25 to 33 percent solids (Table 16).

As the initial settling tests illustrate, the more severe problem in dewatering is with the red mud from digestion of Jamaican bauxite. The settling characteristics of the samples of red mud received from Kaiser's Gramercy refinery and Alcoa's Point Comfort plant appeared to be quite similar. The sample from Alcoa, Point Comfort, was not fully representative as it contained an unexpectedly high percentage of solids (~30.0 percent) and no coarse sand. According to information provided at the plant, the solids content of the mud is generally between 15 and 20 percent and the material contains a sand fraction. Because the experimental activities in this program were of limited scope, detailed dewatering studies were conducted only on the red mud derived from Jamaican bauxite. The term Jamaican Red Mud, as used in the report, refers to the red mud supplied by Kaiser Aluminum Company, Gramercy, Louisiana. The following conditions and operations were studied:

(1) Effect of pH on settling rate

(2) Effect of metal ions on settling rate

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*A very small sample of the mud was provided for the study; therefore, no detailed tests were made.

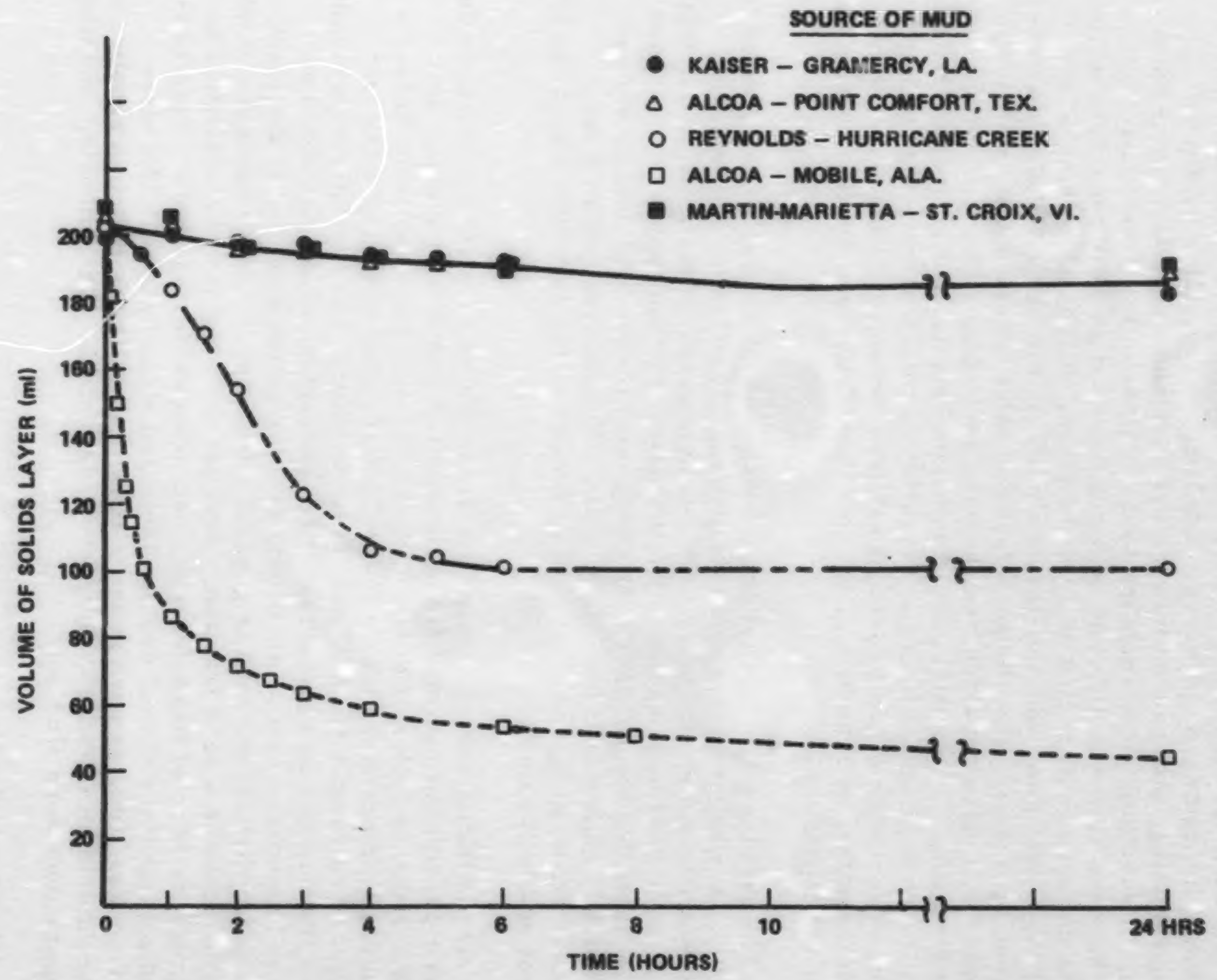

Figure 9. Settling rate curves for various muds (as received)

TABLE 16. SETTLING-RATE DATA FOR VARIOUS MUDS (AS RECEIVED)

Mud source	Type of mud	pH	Settling rate, cm/hr	Initial percent solids in the mud slurry	Percent solids in the sludge after 24-hr settling
Kaiser, Gramercy, Louisiana	Jamaican	11.8	0.10	22.2	25.6
Alcoa, Mobile, Alabama	Surinam-African	11.7	2.96	7.8	28.2
Alcoa, Point Comfort, Texas	Mixed	11.9	0.05	31.0	33.0
Reynolds, Hurricane Creek, Arkansas	Arkansas	11.8	2.15	35.06	55.5
Martin-Marietta, St. Croix, Virgin Islands	Boké	12.3	0.12	35.85	38.04

(3) Effect of polymers on settling

(4) Effect of solids concentration on settling

(5) Effect of magnetic field, i.e., magnetic flocculation, on settling

(6) Flotation as a means to dewater

(7) Centrifuging with and without reagents as a means to dewater.

Effect of pH—The red mud contains different solids with different surface electrical charges. The chemistry of a suspension, particularly the condition of pH can be used to induce a mutual coagulation of mud particles and thereby alter the settling and dewatering character. For this reason, sedimentation experiments were made at different pH.

The settling rate data for the Kaiser-Gramercy (Jamaican) mud at pH 5.2, 7.0, 8.5, and 11.7 are given in Table 17. The pH was not found to have a significant effect on the settling rate. It was noted that adjustment of the pH to below 5.0 caused considerable effervescence and swelling of the mud. No significant change was found in the final solid density of the sludge with pH variation within the 24-hour time period of the experiments.

Effect of metal ions—The fact that metal salts and metal precipitates induce coagulation and settling is well established. Precipitated metal hydroxides enmesh fine particles and accelerate the settling of a suspension.

Several experiments were performed by adding the red-mud slurry to solutions of aluminum (Al^{+3}), ferric (Fe^{+3}), and magnesium (Mg^{+2}) salts. The quantity added was adjusted to give pH values of 8.0, 7.0, and 10.5, respectively, in the final slurry. These pH levels are known to be effective for these metals in industrial wastewater-treatment systems.

An experiment was also made by first lowering the pH of the mud to ~2.0, assuming some of the iron present in the mud might dissolve at this low pH, and then raising the pH to 8.0 to precipitate any dissolved iron as hydroxide. The results are summarized in Table 17. Despite some apparent improvement in the initial rate of settling, particularly in the case of iron, the ultimate settled density of the sludge was not increased above that of the "as-received" sample, or, in the order of 20 to 25 percent solids.

TABLE 17. EFFECT OF pH AND METAL IONS ON SETTLING RATE OF JAMAICAN RED MUD

Volume of red mud, ml	Reagent	Volume of reagent, ml	pH of the mixture	Settling rate, cm/hr	Percent solids in original slurry	Percent solids in sludge after 24 hr
100	H_2SO_4	1.0	5.2	0.188	22.72	23.97
100	H_2SO_4	0.5	7.0	0.072	22.18	23.68
100	H_2SO_4	0.3	8.5	0.085	24.85	25.91
100	--	--	11.8	0.100	24.70	25.46
			• • • • • • •			
75	0.1 M $Al_2(SO_4)_3$	25.0	8.0	0.080	15.03	25.28
50	0.01 M $Al_2(SO_4)_3$	50.0	8.0	0.148	7.25	8.14
75	0.1 M $FeCl_2$	25.0	7.5	0.789	16.94	21.79
75	0.1 M $MgSO_4$	25.0	10.5	0.12	19.56	22.60
100	H_2SO_4 NaOH (1N)	4.0 10.0	8.0	0.075	22.61	24.07

Effect of Synthetic (Polymer) Flocculants

This part of the experimental program was conducted by the Environmental Products Research and Development Group of the Dow Chemical Company under subcontract from Battelle.*

Flocculation due to polymers (or polyelectrolytes) involves adsorption of the polymer with formation of larger aggregated flocs due to a chainlike bridging action. Polymer flocculants are linear or branched high-molecular-weight organic polymers which are water soluble. They are classed according to their ionic character as anionic, cationic, or nonionic, depending on whether their electrical charge is negative, positive, or neutral, respectively.

The flocculation tests made in this program comprised visual observations and optical measurements made when varied quantities of selected flocculants were added to the red-mud sample. Measured volumes of a sample of the suspension were placed in glass containers ("jars") equipped with paddle agitators. Flocculants were then added in measured amounts at prescribed times and agitation conditions. The experimental apparatus comprised six jars arranged as illustrated in Figure 10.

Filtration tests were also made on the flocculated samples. For these tests, the flocculated samples were transferred to a Buchner funnel supported over a graduated cylinder. A record was made of the volume of filtrate collected with time.

In the initial experiments with polymer flocculants, it was noted that the red mud as received formed a gel. The relative effect of different polymers, however, could be determined only by diluting the mud samples.

The initial tests were conducted to establish the proper dilution. These were performed with samples diluted to the following percentage of the as-received samples: 100 (undiluted), 50, 20, 10, 5, and 2 percent. The diluted red-mud suspensions were prepared by dilution with caustic of strength comparable to the original red-mud supernatant. The settling-rate curves for this series of samples are given in Figure 11. It was observed that the 10 percent sample (2.2 percent solids) exhibited a moderate settling rate with a well-defined interface and hence this dilution was selected for all subsequent comparative tests of dewatering agents. The concentration of suspended solids was measured by scattered light and reported in Formazin Turbidity Units (FTU).

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*Dow's report on "Dewatering of Red Mud" is available upon request from the United States Environmental Protection Agency, Cincinnati, Ohio.

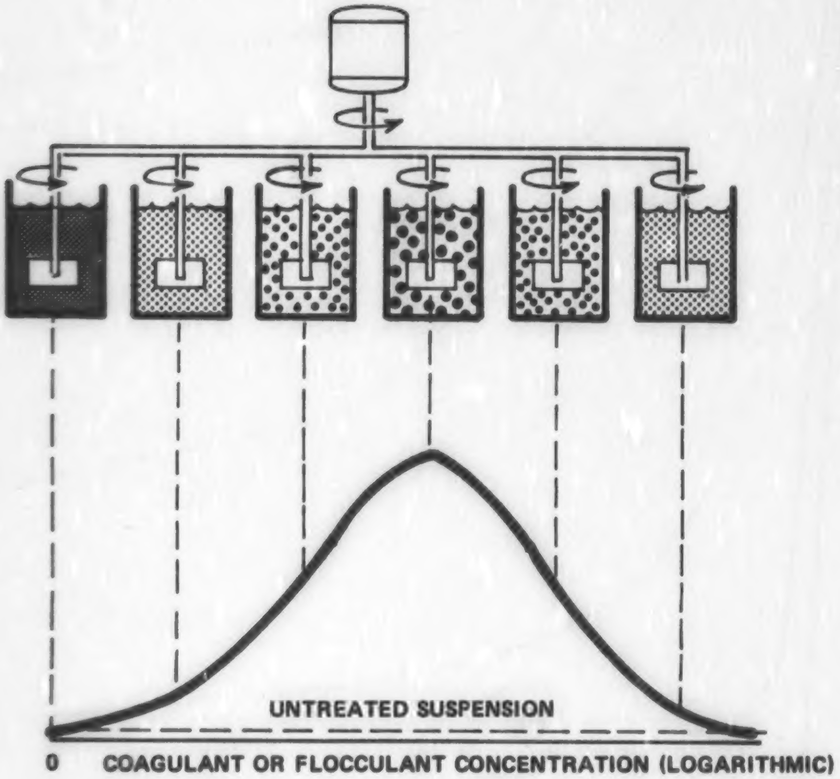
UNTREATED SUSPENSION
0
COAGULANT OR FLOCCULANT CONCENTRATION (LOGARITHMIC)

Figure 10. Laboratory evaluation of efficiency of coagulation – the jar test

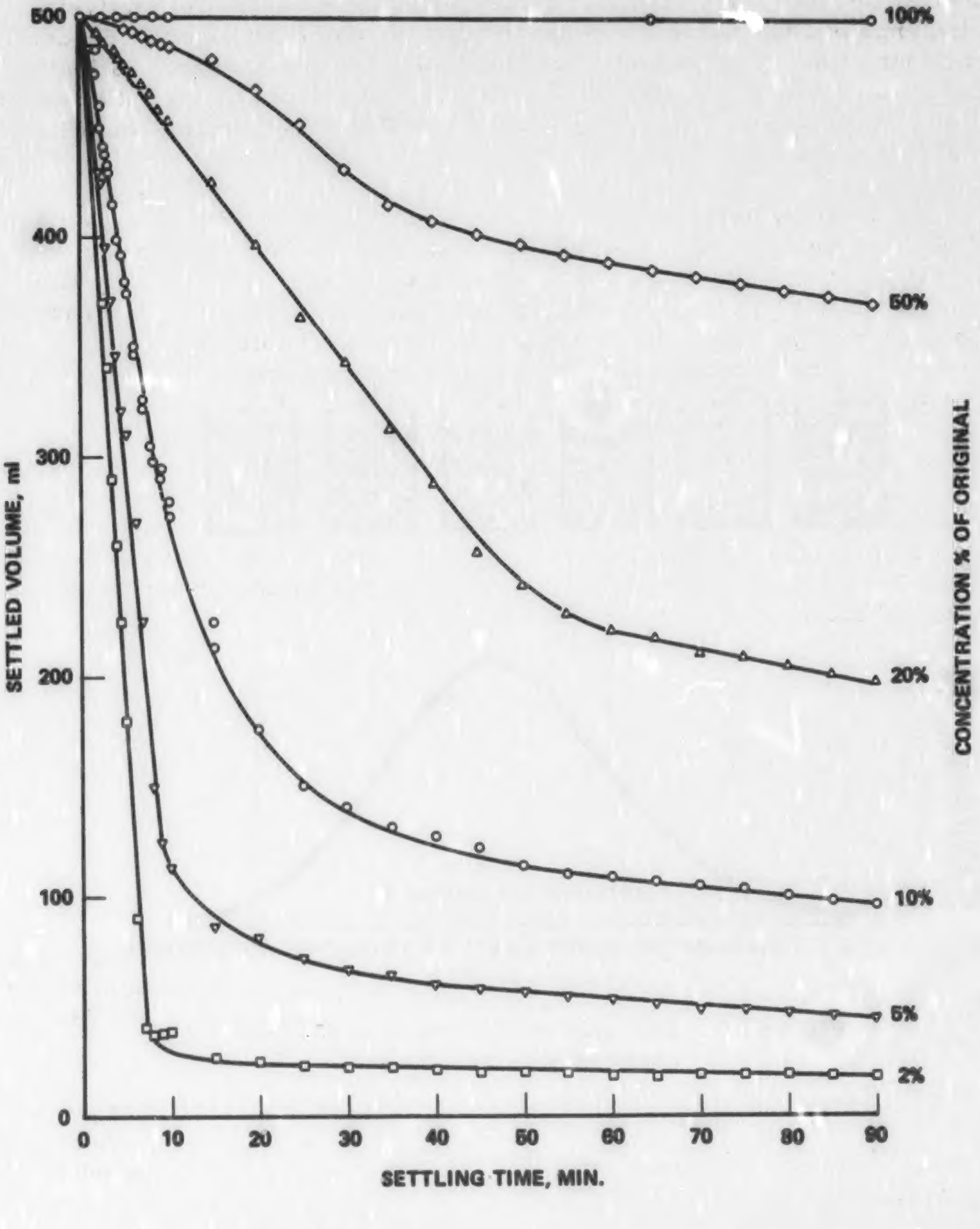

Figure 11. Effect of dilution on settling rate behavior of Jamaican Red Mud

Various synthetic flocculants, listed in Table 18, were tried with the diluted 10 percent red-mud sample. The settling rate and the filtration rate with each flocculant are shown in Figure 12 and Figure 13, respectively. All loadings of flocculants are equal to 0.54 lb/ton of dry solids. Figure 12 shows that anionic and nonionic flocculants are more active than cationic flocculants in effecting settling. The hydrolyzed polyacrylamide (Series III) was most active of all dewatering agents tested for both settling and filtration at this low loading of flocculant.

A few experiments were also conducted at Battelle with undiluted red-mud slurry containing ~22.0 percent solids. The settling-rate experiments using different types of anionic polyelectrolytes indicated that the addition of a synthetic flocculant to the undiluted mud does not significantly improve the settling rate or percent solids of the settled sludge (Table 19).

Magnetic Flocculation

Red mud contains a small amount of free iron and a part of the iron oxide is present as magnetite. A few experiments were conducted to determine whether a magnetic field would affect flocculation. One of the experiments was also conducted with addition of fine magnetite powder (minus 200-mesh size) to the red mud. The results did not show any improvement in the settling behavior or percent solids in the sludge.

Flotation

Flotation of the red mud was performed with two different collectors (an amine and the oleic acid) and frothers (MIBC and cresylic acid). In either case, no segregation of solids was observed in either the froth or the tailings.

Centrifuging

The Kaiser-Gramercy (Jamaican) mud was centrifuged in a laboratory machine at 1000 rpm. The objective was to determine the maximum solids content of a mechanically dewatered mud. Experiments were made with and without addition of a synthetic flocculant (Superfloc C-100).

The centrifuge tubes were 200-ml capacity. These were charged with 100 ml of the mud suspension. Any reagents were added prior to charging. Initial experiments established that compaction was essentially complete after 10 minutes.

The data obtained without reagents and with addition of tallow amine and various amounts of Superfloc C-100 are given in Table 20. The results

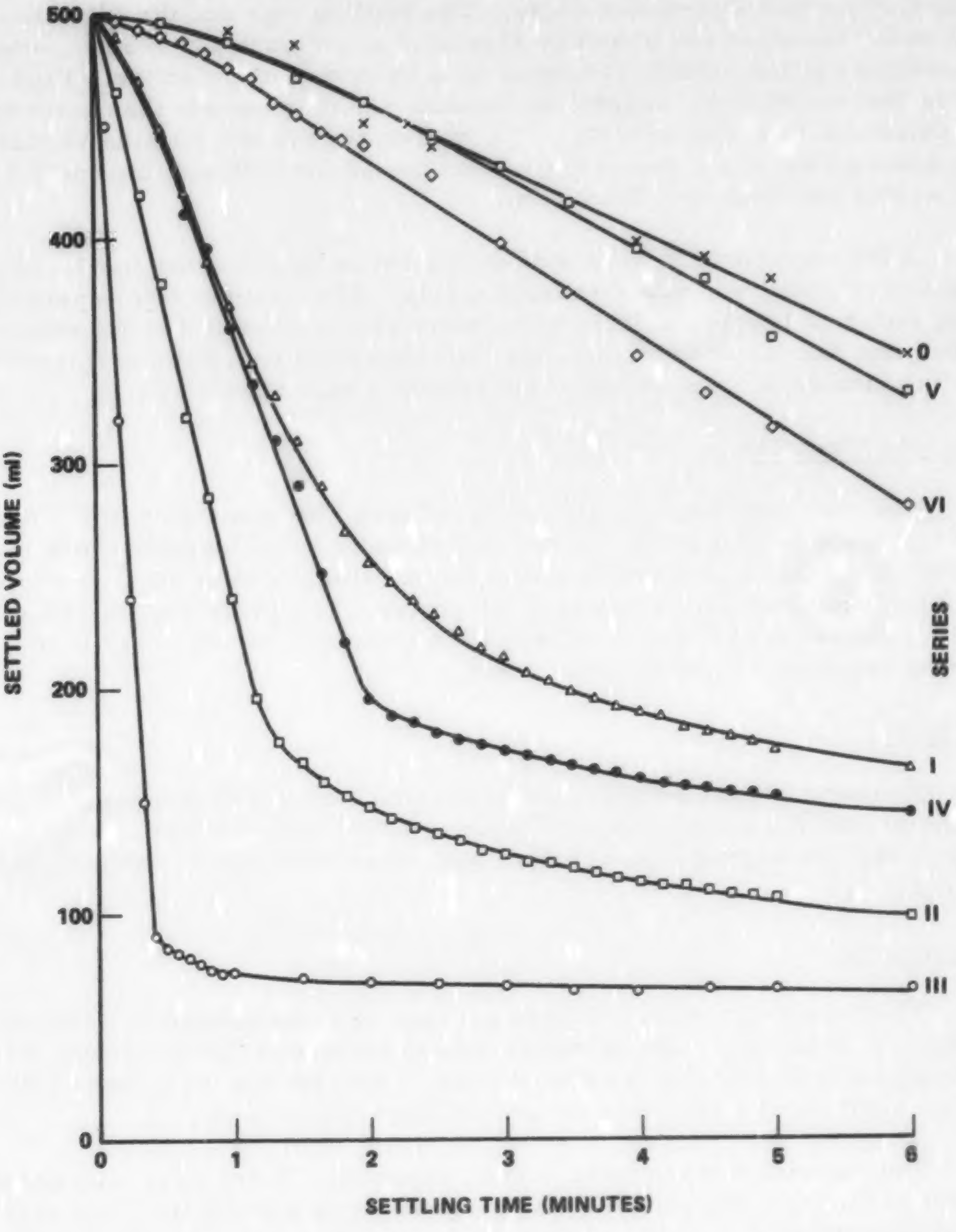

Figure 12. Effect of various synthetic flocculants on the settling rate of diluted (10X) Jamaican Red Mud (Loading of flocculant 0.54 lb/T of solids.)

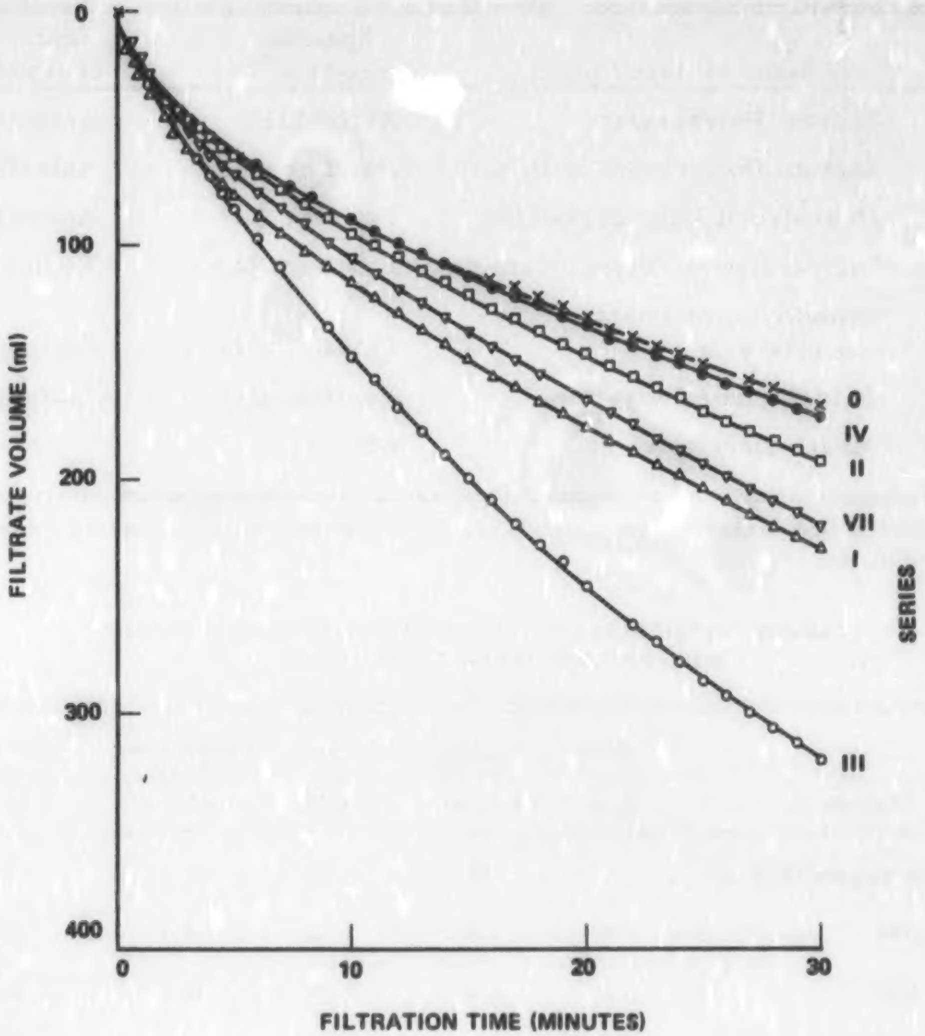

Figure 13. Effect of various synthetic flocculants on filterability of diluted (10X) Jamaican Red Mud (Loading of flocculant 0.54 lb/T of solids.)

TABLE 18. SYNTHETIC FLOCCULANT SELECTED FOR EVALUATION

Series	Chemical description	Specific product[a]	Ionic character
I	Sodium Polyacrylate	XFS-4111L	Anionic
II	Sodium Polystyrene Sulfonate	Purifloc A21	Anionic
III	Hydrolyzed Polyacrylamide	Purifloc A23	Anionic
IV	Unhydrolyzed Polyacrylamide	Purifloc N20	Nonionic
V	Dimethylaminomethyl Polyacrylamide	Purifloc C41	Cationic
VI	Polyalkylene Polyamine	Purifloc C31	Cationic
VII	Starch Derivative	Flocgel	--

(a) All products of Dow Chemical U.S.A. except Flocgel which is produced by W. A. Scholtens Chemische, The Netherlands.

TABLE 19. SETTLING RATE DATA FOR UNDILUTED JAMAICAN RED MUD USING DIFFERENT POLYMER FLOCCULANTS

Polymer	Amount of polymer		Settling rate, cm/hr	Percent solids in original slur:y	Percent solids in sludge after 24 hr
	Ppm	Lb/ton of dry solids			
Control (No Polymer Addition)	--	--	0.03	22.0	25.0
DOW PEI 1090	1	0.09	0.54	17.6	25.8
DOW PEI 1090	5	0.50	0.60	15.1	21.2
DOW PEI 1090	10	0.95	1.20	12.6	17.8
Cyanamid Superfloc C-100	5	0.50	0.16	13.2	28.4
Cyanamid Superfloc C-100	10	0.95	0.10	20.4	23.2
Starch (potato)	100	9.5	0.22	23.0	24.2

show that a dewatered mud of about 40 percent solids is possible by centrifuging. Addition of flocculants did not appear to provide a means to achieve a higher ultimate solids content, thus indicating that only the free water and not the interstitial (bound) water associated with red mud is removed.

TABLE 20. CENTRIFUGE TEST DATA OF JAMAICAN RED MUD[(a)]

Reagent	Volume of reagent, ppm	Initial percent solids in the slurry	Final percent solids in the sludge
--	--	22.80	39.00
Tallow amine	50	21.78	32.00
Superfloc C-100	5	18.75	37.58
Ditto	10	17.98	38.43
"	20	16.72	38.20
"	40	15.24	37.00

(a) Conditions: Centrifuge speed--1000 rpm; time of experiment--10 minutes; volume of mud sample--100 ml.

All of the above studies indicate that additional basic research is needed to better understand the mechanisms which limit or control the removal of interstitial water by physical-chemical methods. This will involve study of surface chemistry of various components of red mud, their affinity for water, and the possibility that the hydrophilic surface properties can be altered.

Reductive Roasting of Bauxite as an Alternative to Conventional Bayer Processing

In the Combination Process, calcination of Bayer Process red mud with soda ash and limestone produces the brown mud (a cement-type material), which is much more readily dewaterable and compacts to a high percent solids. On the basis of this, it was speculated that calcination or sintering of Jamaican bauxite before caustic leaching might alter the clay structure of the ore and yield a different type of red mud.

Technology described by Kamlet(117) in his patent U. S. 2,964,383 assigned to Reynolds Metals Company is of interest not only because it describes a kiln pretreatment before digestion, but it discloses that if the pretreatment is done under a reducing atmosphere condition, it is possible to reduce the iron in the bauxite to a magnetic form, recoverable by usual magnetic separation methods from the leach residue. Several preliminary experiments were made by Battelle using the Kaiser–Gramercy bauxite (Jamaican) ore as the feed to the process. The laboratory procedure comprised the following sequence of steps:

(1) Blending the bauxite ore with sufficient sodium carbonate, calcium carbonate, and carbon. The quantities were based on providing sufficient soda to combine with the alumina to form soluble sodium aluminate, sufficient calcium to combine with silica and titania, and adequate carbon to reduce iron oxides to metallic iron.

(2) Pelletizing the mixture in a balling drum using 10 percent by weight water addition (Experiment 2).

(3) Firing the mixed material (pellets) in a reducing atmosphere at a temperature of 1000 C (1832 F).

(4) Comminuting the calcined product and separating the metallic iron by magnetic separation.

(5) Leaching the residue with an aqueous solution of sodium carbonate to recover the alumina.

The composition used in the experiments was prepared in accordance with the following ratios:

(1) 1.1 moles of Na_2CO_3 added for each 1.0 mole of Al_2O_3
(2) 1.0 mole of $CaCO_3$ added for each 1.0 mole of TiO_2
(3) 2.0 moles of $CaCO_3$ added for each 1.0 mole of SiO_2
(4) Carbon added to amount to 20 percent of the furnace charge.

This part of the experimental program comprised two experiments, which are described below.

Experiment 1 - This experiment was made with a 25-g sample obtained from a ground mixture of bauxite (270 g), sodium carbonate (168 g), calcium carbonate (17 g), and charcoal (121 g). The sample mixture was placed in hollowed-out portion of a 15.24-cm (6 in.)-long and 3.81-cm (1.5 in.)-outside-diameter graphite crucible and heated in an electrically heated tube furnace at 1000 C (1832 F) for 1 hour. The cooled mixture was first leached with 0.1 liter (100 cc) of 10 percent Na_2CO_3 solution and then washed with 1 liter (1000 cc) of hot water. The settling test was performed on the leached residue suspended in water. A Davis tube was used to determine the amount of magnetic iron in the residue. The magnetics and nonmagnetics portions were analyzed separately for iron and Al_2O_3.

Experiment 2 - In the second experiment, pellets 0.64 cm to 1.27 cm (1/4 in. to 1/2 in.) in size were made from the ground bauxite (890 g), sodium carbonate (550 g), calcium carbonate (54.0 g), and coke (200 g) mixture. The pellets were placed in a 11.43-cm (4.5 in.)-long and 7.62-cm

(3 in.)-diameter refractory crucible and fired in an electrically heated muffle furnace at 1000 C (1832 F) for 1-1/2 hours. Fifty grams of the pellets was then crushed, ground, and leached with 200 cc of 10 percent Na_2CO_3 solution. The settling test was performed on the leached solids while suspended in liquor and also in wash water. The Davis tube test was made as before to separate magnetic iron. Analyses of typical products are provided in Tables 21 and 22.

The material balances for the two experiments are listed in Table 23. In the second experiment, emphasis was placed on the settling behavior of solids in pregnant liquor and the leaching of the sample was not completed.

The chemical analyses of the head bauxite sample and other samples obtained at each stage of the sintering process are given in Tables 21 and 22. The overall test data indicated approximately 85 percent recovery of the total available alumina.

Figure 14 shows the settling-rate curves for the mud obtained in the present sinter method test and the red mud obtained in the conventional Bayer Process supplied by the Kaiser plant. Data for the settling rates and percent solids obtained in the sludge for the two muds are given in Table 24.

Note that the settling rate of the mud obtained from the roasted ore experiment is approximately four times higher than that of the conventional red mud. However, the compaction of the mud as measured by the percent solids in the sludge after 24 hours showed no increment in either case.

In general, the preliminary investigations indicated that the soda-lime-sinter process may offer limited potential to reduce the severity of the red-mud problem. The recovery of iron in a magnetic concentrate was only about 30 percent of the iron content of the bauxite. Moreover, the magnetic concentrate was not sufficiently high in iron to be used in ironmaking furnaces. These aspects, however, may be improved with study.

Such a procedure, however, would entail significantly higher energy costs and possibly a need to pelletize the feed to prevent excessive dusting. Moreover, the amount of caustic required may be more than that required for the conventional system. It is not likely that such front-end processing could be implemented without a major change in existing Bayer Process plants.

TABLE 21. CHEMICAL ANALYSES OF PRODUCTS OBTAINED BY THE REDUCTIVE ROASTING OF BAUXITE (EXPERIMENT 1)

Product	Weight percent			
	Al_2O_3	Fe	SiO_2	TiO_2
Head sample (bauxite)	54.10	12.20	0.64	2.44
Sintered mixture	--	8.20	0.98	--
Leached liquor	4.91(a)	--	--	--
Iron concentrate	21.40	41.40	4.40	--
Nonmagnetic residue	10.60	18.70	3.96	3.60

(a) Grams/liter.

TABLE 22. CHEMICAL ANALYSES OF PRODUCTS OBTAINED BY THE REDUCTIVE ROASTING OF BAUXITE (EXPERIMENT 2)

Product	Weight percent			
	Al_2O_3	Fe	SiO_2	TiO_2
Head sample (bauxite)	54.10	12.20	0.64	2.44
Sintered pellets	43.40	11.10	1.36	2.52
Leached liquor	15.78(a)	--	--	--
Iron concentrate	33.90	26.00	1.70	5.30
Nonmagnetic residue	32.30	19.00	1.76	4.21

(a) Grams/liter.

TABLE 23. METALLURGICAL WEIGHT BALANCE FOR IRON AND Al_2O_3 OBTAINED IN REDUCTIVE ROASTING OF BAUXITE

Experiment	Sample	Weight of sample, g	Assays (total weight) Fe, g	Assays (total weight) Al_2O_3, g
I	Head sample mixture(a)	25	2.05	6.27
I	Magnetic concentrate	0.5	0.2	0.10
I	Nonmagnetic concentrate	5.3	1.0	0.57
I	Leached liquor	--	--	5.40
I	Accountability, %		63.0	96.8
I	Recovery, %		10.0	86.0
II	Head sample pellets(b)	50	5.55	21.7
II	Magnetic concentrate	6.4	1.64	2.15
II	Nonmagnetic concentrate	19.2	3.65	6.45
II	Leached liquor	--	--	8.80
II	Accountability, %		95.0	80.0
II	Recovery, %		30.0	40.0

(a) Sample was taken from a 576-g mixture of bauxite (270 g), solution carbonate (168 g), calcium carbonate (17 g), and charcoal (121 g).

(b) Sample was taken from a 1690-g mixture of bauxite (890 g), sodium carbonate (550 g), calcium carbonate (540 g), and coke (200 g) pellets.

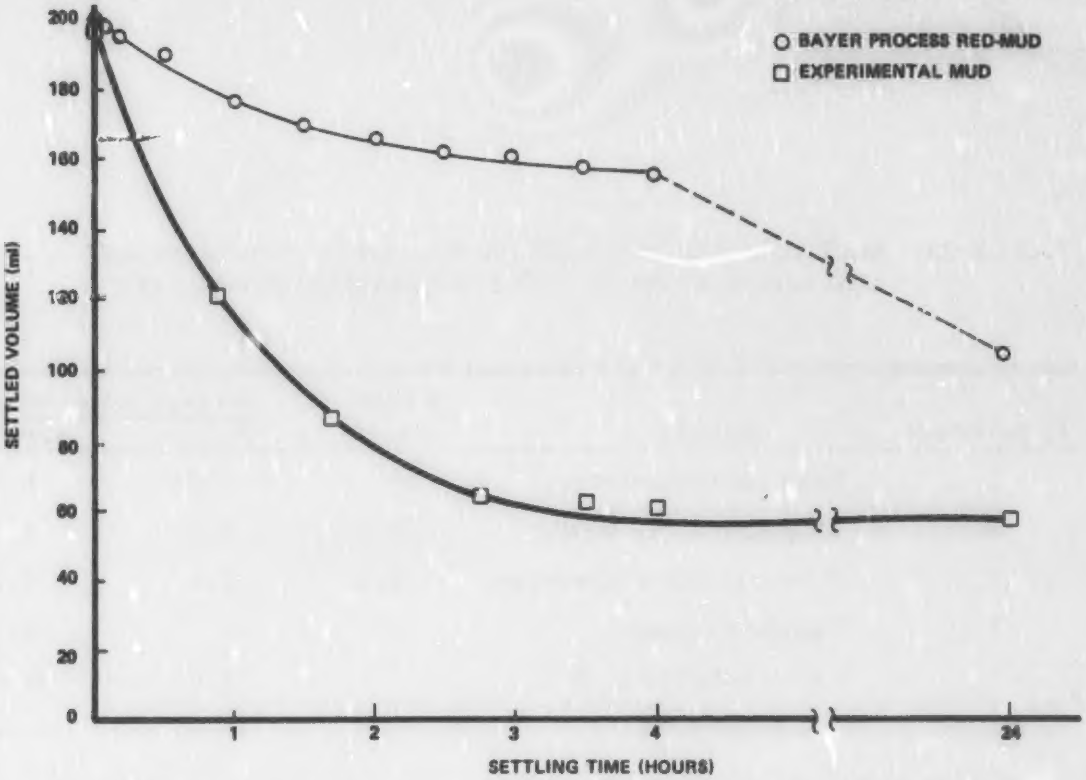

Figure 14. Settling rate curves for the reductive roasting of bauxite process mud (9.7% solids) and the Jamaican Red Mud (11.7% solids) slurries (no flocculant added)

TABLE 24. SETTLING RATE TESTS DATA FOR THE REDUCTIVE ROASTING PRODUCT MUD AND THE JAMAICAN RED MUD

Mud type	Settling rate, cm/hr	Initial percent solids in slurry	Final percent solids in sludge after 24-hr settling
Conventional Bayer Process red mud	3.43	11.71	22.20
Sinter method experimental mud	14.63	9.70	28.50

SECTION VII

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SECTION VIII

GLOSSARY

ALUMINA

Any of several forms of aluminum oxide, Al_2O_3, occurring naturally as corundum, in a hydrated form in bauxite, and with various impurities as ruby, sapphire, and emery.

BAYER PROCESS

Process in which impure alumina in bauxite is dissolved in a hot, strong, alkali solution, normally NaOH, to form sodium aluminate which upon diluting and cooling hydrolyzes, forming a precipitate of pure aluminum hydroxide.

BAUXITE

A commercial ore for alumina, which is an impure mixture of earthy hydrous aluminum oxides and various metal oxides, e.g., iron, titanium, rare earths, etc.

BROWN MUD

The final solid waste remaining after the alumina is leached from the calcined red mud in the combination process.

CALCINATION

The roasting or burning of any substance to bring about physical or chemical changes.

CAUSTIC SODA

The sum of free NaOH and the NaOH combined as $NaAlO_2$, expressed as Na_2CO_3.

CENTIMETER (cm)

0.3937 inch.

CLEAR WATER LAKE

Nominally the lake relatively free of alkalinity and other dissolved solids used as a fresh-water reservoir for a bauxite refinery.

COMBINATION PROCESS

Variation of Bayer Process used for high silica ores, in which the red mud from the first-stage Bayer Process is calcined with soda ash and lime and leached to recover additional alumina.

CONTINUOUS COUNTERCURRENT DECANTATION (CCD)

A continuous system of washing finely divided solids, such as red muds, to free them from liquids containing dissolved substances. In practice, the fresh water and the solids slurry start at opposite ends and move countercurrently to each other, so that the fresh water contacts the solid to remove soluble substances away from it.

DEWATERING

Physical separation of water from sludge by the application of an external force.

DIGESTER

Pressure vessel or autoclave in which the alumina is dissolved from the bauxite.

FRENCH DRAIN

An underground passageway (through gravity) for liquid through the interstices of sand and gravel layer and collected in perforated pipes.

FILTER CAKE

The solids collected on a mechanical filter surface when a solids suspension is filtered.

FLOC

Collections of smaller particles agglomerated into larger, more easily settleable particles through chemical or physical treatment.

FLOCCULANT

A reagent which induces flocculation of suspended solids.

FLOCCULATION

The agglomeration of suspended solids by adding a flocculant, thereby forming larger aggregate flocs that are easily removed by sedimentation.

FLOTATION

A process for separating suspended particles from a solution by introducing air bubbles which carry the particles to the surface where they are removed.

GPM

Gallons per minute.

GREEN LIQUOR

The aluminum-bearing solution from the bauxite digesters before further processing.

HINDERED SETTLING (Sedimentation)

Sedimentation that is affected by interactions among suspended particles at high concentrations or between particles and vessel walls.

IMPOUNDMENT

A natural or artificial, usually open, body of water from which surface water could be reused.

JAR TEST

A laboratory procedure for evaluating flocculation and sedimentation processes in a series of parallel comparisons.

LITER

1000 cubic centimeter.

MAGNETIC SEPARATION

A physical treatment process for removing magnetic suspended solids from a liquid by applying a magnetic field.

MICRON

0.0001 cm (10^{-6} meter).

mg/l

Milligrams per liter. Nearly equivalent to parts per million concentration.

MUD LAKE

The diked reservoir (tailing pond) used to impound mud.

pH

A measure of the alkalinity or acidity of a solution, in terms of the negative logarithm of the hydrogen-ion $<H^+>$ concentration (mol/l); neutral = pH 7 = 10^{-7} mol $<H^+>$/l; acidic pH < 7; basic pH > 7.

POLYMER

Any synthetic organic compound having a high molecular weight and composed of repeating chemical units (monomer).

ppm

Parts per million, a unit of concentration.

PRECIPITATION

The conversion of dissolved solids into suspended solids.

PREGNANT LIQUOR

Solution containing the metal values prior to their removal and recovery.

PROCESS LAKE

Reservoir used for process water; often in closed circuit with part of process; not used for mud disposal.

RECYCLE

To return water after some type of treatment for further use, generally implying a closed system.

RED MUD

The final solid waste remaining after the alumina is leached from the bauxite.

SAND FILTER

A bed of sand through which liquid is passed to remove fine suspended particles from it.

SEDIMENT

Any solid phase settling out of a liquid phase.

SEDIMENTATION

Solid-liquid separation resulting from the application of an external force, usually under the force of gravity.

SLUDGE

Any solid material containing entrained water.

SLURRY

Suspension of solids in a liquid.

SUPERNATANT

The liquid remaining above the settled sludge after sedimentation.

TURBIDITY

Any suspended solids imparting a visible haze or cloudiness to water.

APPENDIX A: LITERATURE REFERENCES BY CATEGORY

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APPENDIX B: WORKSHOP SESSIONS

Draft report copies of the project were submitted for review to the U. S. Environmental Protection Agency and aluminum industries of the United States in the month of January, 1976. A workshop on the project was sponsored by the U. S. Environmental Protection Agency. The workshop sessions were held at Battelle on March 30 and 31, 1976, to discuss and receive comments on the report. The workshop was attended by representatives of the U. S. Environmental Protection Agency, four domestic aluminum companies, and Dow Chemical Company. The program and list of the attendees with their title and organization are listed in Table B-1 and Table B-2, respectively.

The present report incorporates many of the comments and recommendations made by the various representatives during the workshop sessions.

TABLE B-1. PROGRAM OF THE WORKSHOP

Tuesday, March 30, 1976	
9:00 a.m.	Welcome to Battelle
9:10 a.m.	Arrangements for Workshop
9:20 a.m.	Introductory Comments (EPA, Battelle)
9:30 a.m.	Review of the Project and Draft Report (Battelle, Dow)
10:30 a.m.	Coffee Break
10:45 a.m.	Prepared General Comments by Industry Participants
12:15 p.m.	Lunch
1:30 p.m.	Workshop Discussions
	Topics: Processing, Disposal, and Utilization
3:00 p.m.	Coffee Break
3:15 p.m.	Demonstration of Advanced Information Processing and Retrieval Systems
3:45 p.m.	Continue Workshop Discussion
4:30 p.m.	Adjourn
Wednesday, March 31, 1976	
9:00 a.m.	Recommended Research and Demonstration Projects (Industry Participants)
10:45 a.m.	Summary of Workshop Results (Battelle)
11:15 a.m.	Concluding Remarks by Participants

TABLE B-2. LIST OF THE WORKSHOP ATTENDEES*

Organization	Name	Title
Kaiser Aluminum[(a)]	Mr. A. E. McLaughlin	Manager, Process Development
Kaiser Aluminum	Dr. R. M. Hansen	Environmental Manager
Alcoa[(b)]	Mr. J. E. Ralphe	Superintendent – New Project
Alcoa	Mr. R. H. Carwile	Environmental Control Engineer
Reynolds Metals Company	Mr. M. Handelman	Technical Assistant to Plant Manager
Reynolds Metals Company	Mr. F. N. Newchurch	Process Control Leader
Reynolds Metals Company	Mr. J. T. Robinson	Senior Process Engineer
Reynolds Metals Company	Mr. R. N. Reid	Senior Environmental Engineer
Martin-Marietta	Mr. J.A.S. Green	Head, Materials Department
U. S. EPA[(c)]	Mr. Donald Wilson	Research Chemist
U. S. EPA	Mr. T. G. Newport	
Dow Chemical Company	Dr. S. L. Daniels	Research Specialist

*List does not include names of Battelle's participants.

(a) Kaiser Aluminum and Chemical Corporation
(b) Aluminum Company of America
(c) U. S. Environmental Protection Agency